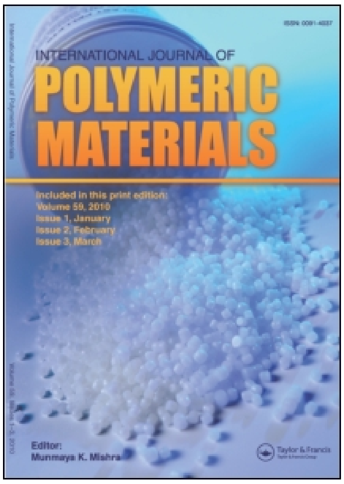

**International Journal of Polymeric Materials**
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713647664

# Thermo-oxidative aging and fatigue behavior of dynamically vulcanized PVC/ENR thermoplastic elastomers

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**To cite this Article** Mousa, A. , Ishiaku, U. S. and Ishak, Z. A. Mohd(2002) 'Thermo-oxidative aging and fatigue behavior of dynamically vulcanized PVC/ENR thermoplastic elastomers', International Journal of Polymeric Materials, 51: 11, 967 — 980

**To link to this Article: DOI:** 10.1080/714975687

**URL:** http://dx.doi.org/10.1080/714975687

*International Journal of Polymeric Materials*, 51:967–980, 2002

0091-4037/02 $12.00 + .00
DOI: 10.1080/00914030290109813

# THERMO-OXIDATIVE AGING AND FATIGUE BEHAVIOR OF DYNAMICALLY VULCANIZED PVC/ENR THERMOPLASTIC ELASTOMERS

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*Dynamically vulcanized poly(vinyl chloride)/epoxidized natural rubber (PVC/ENR) thermoplastic elastomers (TPEs) were prepared following a semi-efficient vulcanization system (semi-EV) with the incorporation of di-ethylhexyl phthalate (DOP) as a plasticizer. The PVC/ENR TPEs were melt mixed at 150°C and 50 rpm using a Brabender Plasticorder. The effect of dynamic curing on the fatigue behavior of the PVC/ENR TPEs was investigated. The properties investigated include fatigue life, stress – strain behavior, strain energy, morphology, and the effect of hysteresis. The unaged samples showed that as the sulfur concentration increases the fatigue life increases which could be related to the increase in cross-link density as indicated from the swelling index data. The increase in cross-link density increases the resistance of these materials against external dynamic stress. Hysteresis study revealed that samples with higher sulfur concentration are less sensitive towards changes in strain energy. As a result they exhibited lower heat build-up and consequently higher fatigue life. The effect of thermo-oxidative aging (TOA) on the fatigue behavior was investigated by exposing the PVC/ENR TPEs in an air oven at 80°C for 168 hours. It was found that all the properties studied decreased after thermo-oxidative aging process. This suggests that some micro-structural changes such as the formation of new cross-links due to post curing has taken place. This accounts for the reduced fatigue life and resilience while hysteresis, modulus, and hardness increased.*

*Keywords*: fatigue life, thermoplastic elastomers, hysteresis, aging

## INTRODUCTION

Thermoplastic elastomers (TPEs) are polymers that combine the processibility of plastomers and the functional performance of the conventional elastomers i.e., chemically cross-linked rubbers. There are two major groups

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Received 19 June 2000; in final form 24 June 2000.

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of commercially available TPEs: block copolymers and elastomeric alloys. Elastomeric alloys are blends of a rubber and plastic in which the rubber component has been dynamically vulcanized. Dynamic vulcanization is the process of effecting vulcanization during the mixing stage with the thermoplastic. It is theoretically possible to produce great number of elastomeric alloys such as PVC/ENR blends. Miscibility studies of PVC/ENR blends revealed that the two polymers are miscible [1 – 3]. Di-ethylhexyl phthalte (DOP) plasticized PVC/ENR blend has been reported to be a miscible blend with a single Tg [4]. Plasticized PVC/ENR blend can be classified as a thermoplastic elastomer, which looks, feels and performs like vulcanized rubber [5]. In a related study Mousa et al. [6] studied the oil resistance of the dynamically vulcanized PVC/ENR TPEs. They found that dynamic vulcanization imparted excellent retention of the tensile properties upon immersion in ASTM oil # 3 at above ambient temperature i.e., 100°C. The oil resistance of the plasticized PVC/ENR was within the range of plasticized PVC/NBR as demonstrated by Mousa [7]. In another related study Mousa et al. [8] reported on the effect of dynamic vulcanization on the mixing rheology of the dynamically vulcanized PVC/ENR. They found that as the sulfur content increased the mixing torque was increased which is an indirect indication of the cross-link density. A major source of failure in rubber products under prolonged dynamic service conditions is the development of cracks either within the rubber or at the surface. On repeated deformations these cracks can grow and lead to a complete failure. Fatigue is one of the most important long term test methods for both natural rubber and synthetic rubber [9]. The results of the standard fatigue test do not usually correlate with the service performance of the compound because the manner of flex cracking depends on the geometry of the part the type of stressing and the environmental conditions [10]. Recently Ishiaku et al. [4, 11, 12], found that the introduction of plasticizers such as DOP and etherthioether have successfully curbed degradation of the PVC/ENR and the plasticized blend showed thermo-oxidative stability. In this article emphasis is placed on the effect of cross-linking *via* dynamic vulcanization and the effect of thermo-oxidative aging on the fatigue properties of PVC/ENR TPEs. This is of interest because to date no fatigue study on PVC/ENR TPEs was published according to the authors knowledge. So far most of the dynamically vulcanized PVC/ENR TPEs properties involve short term properties such as tensile properties and tear strengths while the number of long term properties is limited. Thus, the present work is a step further in the effort to characterize the mechanical properties and thermo-oxidative aging of PVC/ENR TPEs. It is quite obvious that such properties are valuable in predicting the service behavior of the materials. In the present study we report further attempts to improve the mechanical properties and the aging behavior of the plasticized PVC/ENR blends *via* the incorporation of curatives.

# EXPERIMENTAL

## Materials

Suspension polymerized PVC in powder form, with a K-value of 65 and a degree of polymerization of 920 – 1060 was supplied by Malayan Electro Chemical Industry Sdn Bhd, Penang, Malaysia. ENR with 50 mole % epoxidation (ENR-50) was obtained from Kumpulan Guthrie Bhd Seremban, Malaysia. Lead stearate $Pb(St)_2$ was obtained from Komita Sdn Bhd, Malaysia. DOP, sulfur, tetramethylthiuram disulfide (TMTD), 2,2-dithiobis benzothiazole (MBTS), zinc oxide, stearic acid were obtained from Bayer (M) Sdn Bhd, Malaysia.

## Formulations

A semi- efficient sulfur vulcanization system (semi-EV) was incorporated into the plasticized PVC/ENR blend. The formulation used in this study is given in Table 1.

## Melt Mixing

PVC was initially premixed with 3 phr lead stabilizer in a Janke and Kunkel IKA Labotechnik dry mixer at 300 rpm for 10 min at 30°C. Melt mixing was performed using a Brabender Plasticorder Model PLE 331 coupled with mixer/measuring head (W 50 H) at 150°C and 50 rpm rotor speed [2]. The ENR-50 was charged into the mixing chamber to equilibrate, followed by PVC, zinc oxide and stearic acid. After sufficient melt mixing of the PVC the accelerators, TMTD and MBTS, were added. Mixing was allowed to proceed for 8 min after which the sulfur was added. Curing then occurred and was indicated by an increase in mixing torque with increase in sulfur concentration. Mixing was then continued at a reduced speed of 30 rpm until a constant torque was obtained [13]. The compound was removed from the mixer and sheeted on a cold two roll mill. It was passed once through the

**TABLE 1** Recipe used to produce PVC/ENR TPEs

| | |
|---|---|
| PVC | 70 parts by weight |
| ENR-50 | 30 parts by weight |
| DOP | 50 phr PVC |
| Zinc oxide | 3 phr ENR |
| Stearic acid | 1.5 phr ENR |
| Sulfur | X |
| MBTS | X |
| TMTD | 1/3 X |

Where X is the amount of sulfur was varied from 0 – 1 phr ENR-50.

nip to produce approximately a 1 mm thick sheet. The sheets were cut into strips and again subjected to Brabender mixing at 150°C and 50 rpm for 2 min to assure homogeneous melt after which it was again sheeted out prior to compression molding [6, 8, 13].

## Molding

Compounds were compression molded at 150°C for 15 min with a pressure of 10 MPa into rectangular sheets ($22.9 \times 7.6 \times 0.15\,\text{cm}^3$) with beaded edges. Individual dumbbell samples were cut at right angles using a BS type E dumbbell cutter.

## Fatigue Life

Fatigue tests of the vulcanizates were carried out on a Monsanto Fatigue to Failure Tester (FTFT). Six samples of each compound had been cut and tested at different extension ratios ranging from 1.7 – 1.9. The number of cycles were recorded automatically. The fatigue life in kilocycles was computed as J.I.S. average, which was determined from the highest four values using the formula:

$$\text{J.I.S. average} = 0.5A + 0.3B + 0.1(C + D) \tag{1}$$

where $A$ is the highest value followed by $B$, $C$ and $D$.

## Strain Energy Measurement

Fatigue specimen was cycled for more than 30 cycles by the FTFT at the maximum extension ratio which used to determine the fatigue life curve prior to strain energy measurement. The specimen was clamped to a strain energy equipment that consists of a stand incorporating lower clamp (weight 50 g). The width and the thickness of the samples were measured using a dial gauge. Horizontal chalk lines were drawn on the samples in the linear region when fully extended. The pan was then detached and the unloaded distance between the marks were measured. Distances were also measured by replacing the pan with 50 g weight, followed by successive additional loading of 200 g at one minute interval. The load/extension ratio results was processed manually and plotted against the extension ratio. The stress values were read off from the curves at an interval of 0.1 extension ratio. Strain energy was obtained by using Simpson's rule to sets of three consecutive results to give the areas ($A$) of sections of the curves and subsequently the total area of the curves at extension ratio increment of 0.2 as in the following equation:

$$\text{Area} = \frac{1}{3}a[h_x + 4h_{(x-1)} + h_{(x+2)}] \text{ where } a = 0.1 \text{ (strain interval)} \tag{2}$$

## Resilience

Resilience test was carried using Dunlop Pendulum according to BS 903, Part A8 and the rebound resilience was calculated as follows:

$$\text{Resilience}(R) = \frac{1 - \cos(\text{angle of rebound})}{1 - \cos(\text{angle of fall})} \tag{3}$$

The energy not recovered is hysteresis and appears as heat. Hysteresis = 100 – resilience.

## Swelling Index

Specimens in the form of 2 mm thick circular discs with diameters 40 mm were immersed in toluene for 12 hours and the diameters of the swollen samples were measured. Swelling index was calculated as follows:

$$\text{Swelling Index} = \frac{\text{Swollen Mass}}{\text{Original Mass}} \tag{4}$$

## Thermo-Oxidative Aging

Thermo-oxidative aging studies were performed according to BS 7646. The samples were placed in an air oven and aged at 80°C for 168 hours. Retention of properties was calculated as shown below:

$$\text{Retention\%} = \frac{\text{Value after aging}}{\text{Value before aging}} \times 100 \tag{5}$$

## Hardness Test

The Wallace Dead Load Hardness tester was used to measure hardness in an International Rubber Hardness Degree (IRHD). The procedure was used according to ISO 48 (1979).

## RESULTS AND DISCUSSION

### Effect of Sulfur Concentration on Fatigue Behavior

### *Fatigue Life*

Figure 1 shows a plot of fatigue life *versus* extension ratio for PVC/ENR TPEs containing various loading of sulfur. At any extension ratio, the incorporation of sulfur has resulted in an increase in the fatigue life of PVC/ENR TPEs. This is most likely due to the action of the curing system which results in the formation of a three dimensional network structure within the

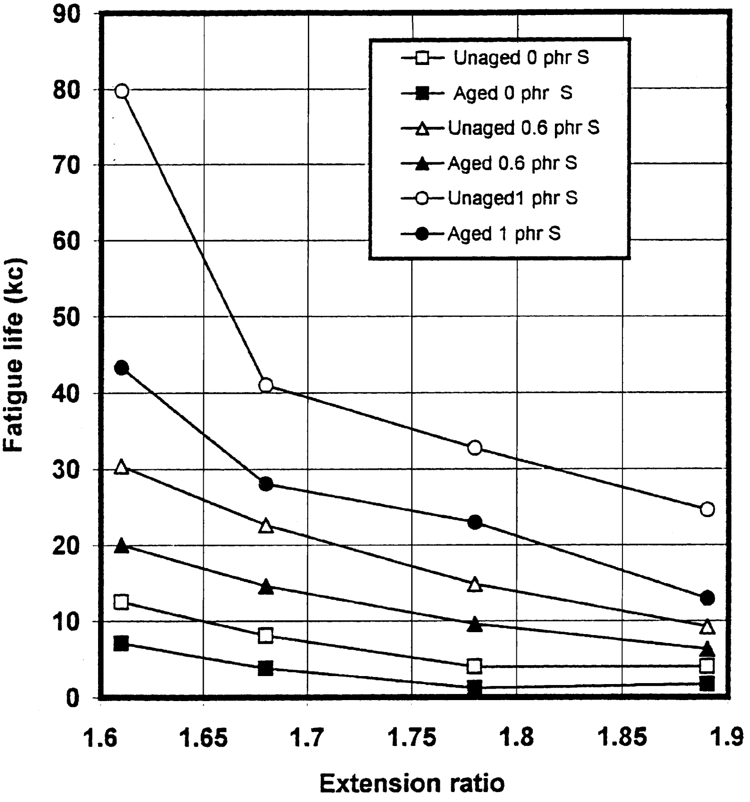

**FIGURE 1** The effect of sulfur loading on the fatigue life of unaged and aged PVC/ENR TPEs.

ENR. The increase in the fatigue life of PVC/ENR TPEs with sulfur loading can be attributed to the increase in cross-link density in the dispersed rubber particles. Swelling index data shown in Table 2 provides a good indication to the extent of cross-linking, as the decrease in swelling is an inverse function of the increase in cross-linking. The increase in cross-link density with the increase in sulfur dosages through dynamic vulcanization process is expected to increase the elasticity of the cross-linked rubber particles in the PVC/ENR TPEs. This consequently lead to the increase of the rebound resilience as shown in Figure 2. This implies that the elasticity of the network upon the cyclic deformation has increased with the increases in sulfur dosage. Thus, the more elastic the chain is, the higher the resilience it has towards cyclic deformation, hence a higher fatigue life is expected. The effect of network chain mobility on the fatigue life of rubber vulcanizate has also been reported by Cox and Parks [14]. In the case of filled ENR

**TABLE 2** The effect of sulfur loading on the swelling index of the aged and unaged PVC/ENR TPEs

| *Sulfur content (phr)* | *Unaged PVC/ENR* | *Aged PVC/ENR* |
|---|---|---|
| 0 | 1.88 | 1.56 |
| 0.2 | 1.84 | 1.48 |
| 0.4 | 1.73 | 1.38 |
| 0.6 | 1.70 | 1.36 |
| 0.8 | 1.68 | 1.34 |
| 1.0 | 1.46 | 1.27 |

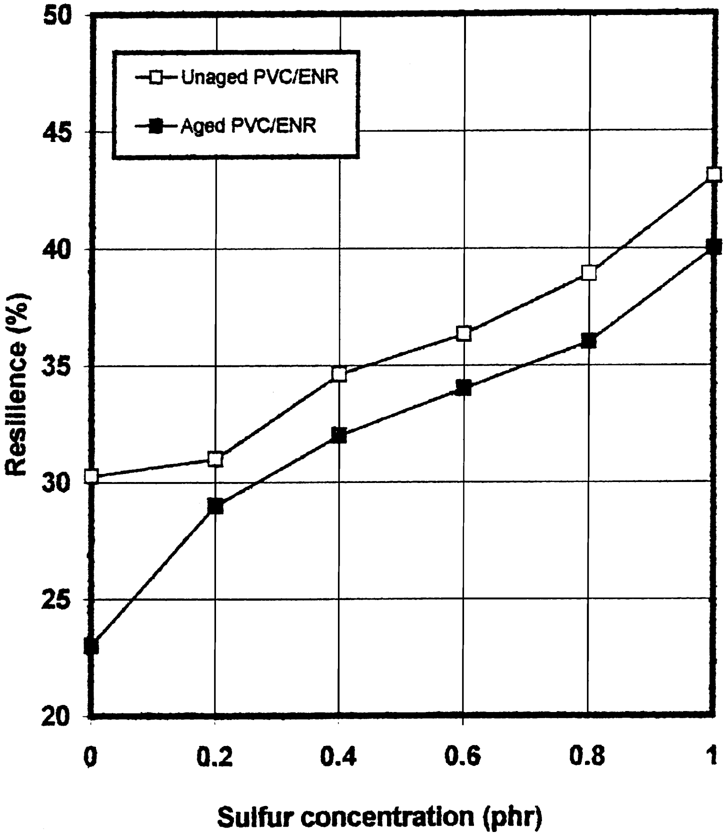

**FIGURE 2** The effect of sulfur concentration on the rebound resilience of unaged and aged PVC/ENR TPEs.

vulcanizates Mohd. Ishak et al. [15] have reported that the types cross-linking in the rubber vulcanizates are also capable of influencing the fatigue life. Vulcanizates with polysulphidic cross-links have been proven to be more flexible and more resistance to cyclic deformation compared to mono-sulphidic cross-links. This has been attributed to the efficiency of the long cross-links in distributing the stress uniformly in the vulcanizates. Figure 1 also, illustrates the variation of fatigue life with respect to changes in extension ratio. It can be seen that, irrespective of sulfur loading, the fatigue life of PVC/ENR TPEs samples diminish as the extension ratio increases. This could be attributed to the increase in tearing energy as the strain increases. Thus, the rate of cut growth will be accelerated towards lower fatigue life. This is in agreement with data presented in Figure 3 which will

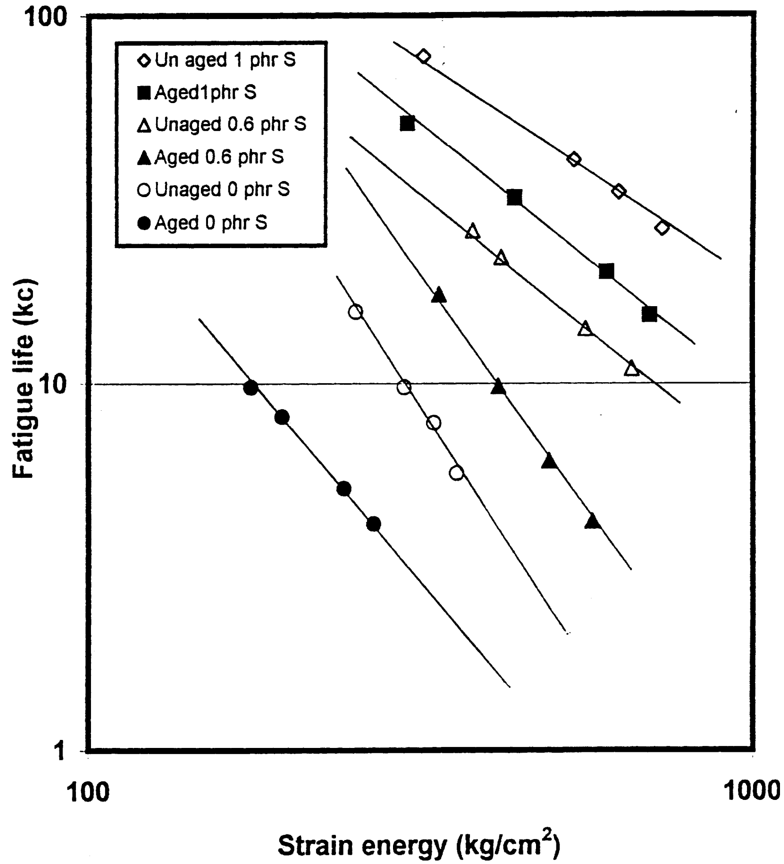

FIGURE 3 The influence of accumulative strain energy on the fatigue life of unaged and aged PVC/ENR TPEs.

be detailed in the following section. The current data are in line with the trend of the major effects of vulcanization on the vulcanizate structure as mentioned by several workers [15–17].

## Effect of Hysteresis

Hysteresis is an important aspect when considering the fatigue properties of elastomers. It is a measure of the energy loss which lead to heat build-up when materials are subjected to dynamic deformation. Consequently, the fatigue life could be shortened. According to Gent [18] fatigue life is a function of strain energy density which is the same as the concept of tearing energy. At higher strain energy density, i.e., high strains, the flex life decreases. Figure 3 depicts the effect of strain energy on the fatigue life at various sulfur loading of PVC/ENR TPEs. The hysteresis value is indicated by the strain exponent $n$ which can be calculated from the slope of log – log graphs of the fatigue life against the strain energy using linear regression method. The strain exponent $n$ is related to the number of cycles to failure, $N$ as [19]:

$$N = \frac{G'}{(n-1)WC_o^{n-1}} \tag{6}$$

$G'$ = modified cut growth constant.
$C_o$ = effective critical flaw size.
$W$ = strain energy per unit volume.

Figure 4 illustrates the influence of sulfur concentration on the strain energy exponent $n$ for PVC/ENR TPEs. The higher the $n$ value, the greater is the sensitivity of fatigue life of the PVC/ENR TPEs towards changes in strain energy, as exhibited by the uncrosslinked system. At very low crosslink density, it is expected that the resilience will become independent of the structure of the vulcanizate as observed by Bristow and Tiller [20]. The rebound resilience improved to some extent as the degree of crosslinking rises as elaborated in the previous section and presented in Figure 2. Therefore, this implies that the PVC/ENR sample with the lower sulfur content is more sensitive toward changes in strain energy. Thus, it exhibited higher hysteresis (shown by the $n$ values) and consequently higher heat build-up. This will eventually shorten the fatigue life of these materials. This trend is in agreement with the values of fatigue life of the respective TPEs given in Figure 1. In a relevant study on the rheological and viscoelastic behavior of PVC/ENR TPEs, Mousa et al. [21] found that as the sulfur dosage increased the extent of heat loss or damping has been reduced, i.e., a less hysteretic compound with increased rebound resilience and reduced heat build up. This observation also agrees quite well with the

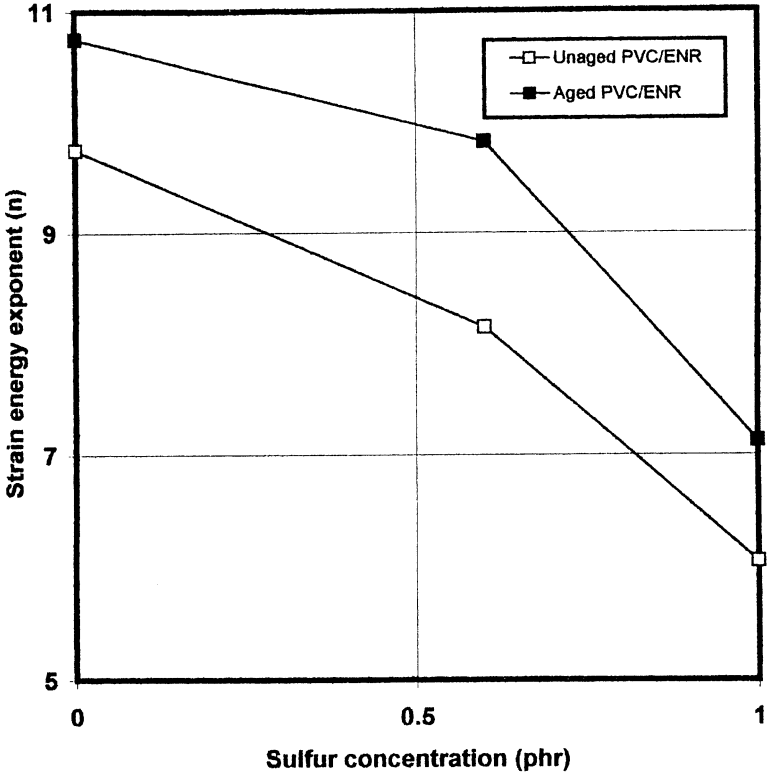

**FIGURE 4** The influence of sulfur concentration on strain energy exponent of unaged and aged PVC/ENR TPEs.

previous investigation by Mohd. Ishak et al. [15] on the effect of hysteresis on ENR-50 filled with different loadings of silica and carbon black.

### *Stress – Strain Curves*

Figure 5 shows the stress – strain behavior of the PVC/ENR TPEs. It can be seen that, the stress increases with increasing in sulfur loading at any extension ratio. Since modulus is a direct function of cross-link formation, it can be inferred that cross-link density increases with increase in sulfur loading. This again correlates well with the swelling index data shown in Table 2. It is believed that the formation of intermolecular network through dynamic vulcanization process produces a stiffer structure of PVC/ENR TPEs which requires higher external stress to fail. In addition the cross-link formation enhances the resilience of the PVC/ENR TPEs which increases the resistance of the sample against cyclic deformation.

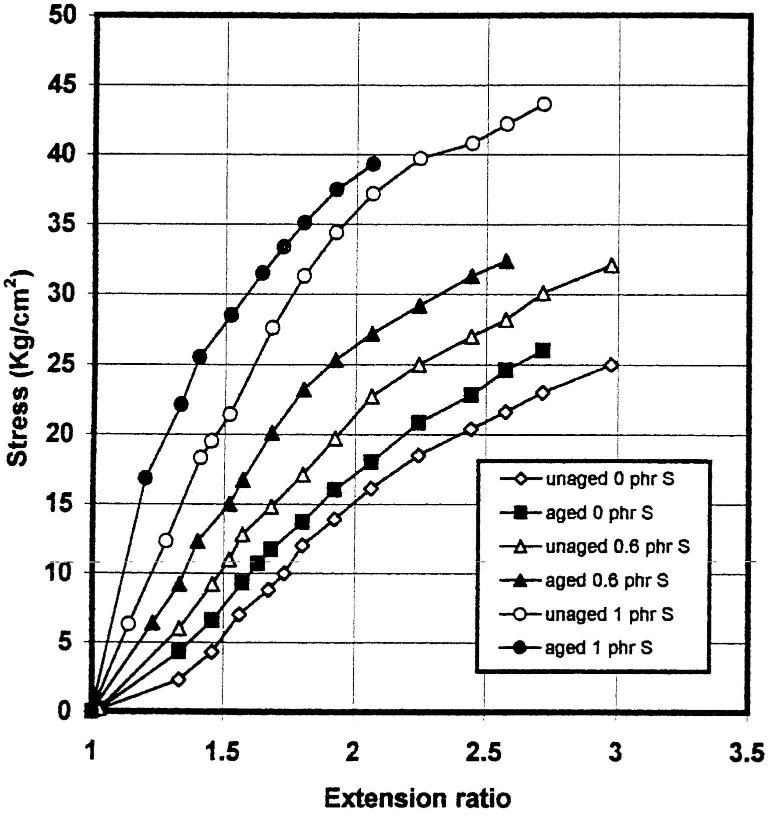

FIGURE 5 Stress–extension ratio curve of PVC/ENR TPEs.

## Effect of Thermo-Oxidative Aging on Fatigue Behavior

In the preceding section it has been shown that the cross-link formation within the ENR after the addition of the curatives has resulted in significant changes in the fatigue behavior of the TPEs. In the following section the effect of thermo-oxidative aging (TOA) on the fatigue behavior is discussed.

### Fatigue Life

The effect of TOA on fatigue life of PVC/ENR TPEs is also shown in Figure 1. Again, irrespective of sulfur loading, the fatigue life of the thermo-oxidatively aged samples reduced with increasing extension ratio. The exposure to high temperature for a prolonged period has remarkably decreased the fatigue life of the dynamically vulcanized PVC/ENR TPEs.

This is a typical characteristic of post curing which arises from the formation of more sulfur cross-links due to the aging process. As mentioned earlier, fatigue life is related to the stiffness and resilience of the rubber chain in the vulcanizate. The increase in cross-link density as indicated from the swelling data in Table 2, will increase the stiffness of the aged PVC/ENR TPEs and decrease the flexibility. Thus, this explains the reduction in the rebound resilience of the thermooxidatively aged samples as shown in Figure 2. According to Hoffman [22], the formation of new cross-links will shorten the polythioether bridges. The reduction in the flexibility of the cross-links will affect the stress distributions in the vulcanizates and also limit its ability to relieve the stresses. This is also in agreement with a recent work reported by Hanafi et al. [23] on filled ENR compounds. Figure 5 shows the stress – strain behavior of the aged PVC/ENR TPEs. It is obvious that the 1 phr sulfur loading exhibited the higher stress at any extension ratio. Cross-links formation effectively reduces mobility, and thus accounts for the increase in modulus of the aged samples as compared to the unaged ones. This result is further supported by hardening of the samples after TOA as presented in Figure 6. The reduction in the extension ratio (strain) could be attributed to the shorter and therefore more rigid network, this consequently leads to a lowering of fatigue life. This suggests that the work required to deform the PVC/ENR specimen is higher for the cross-linked system.

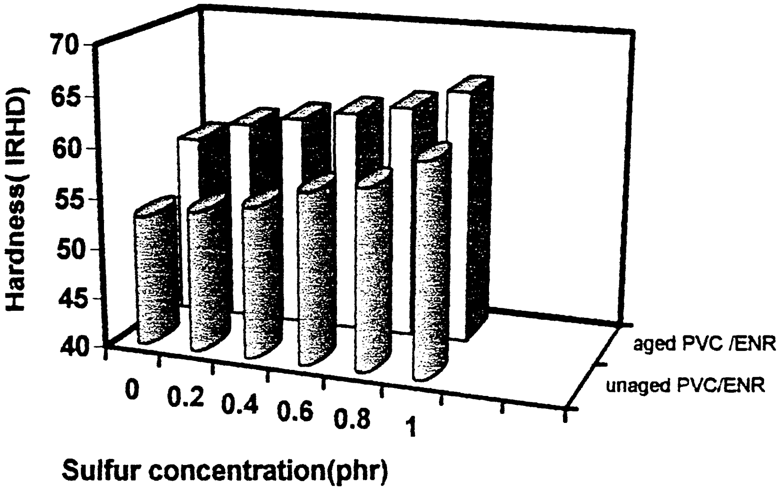

FIGURE 6 The effect of sulfur loading on the hardness of unaged and aged PVC/ENR TPEs.

### Effect of Hysteresis

Figure 3 shows the relationship between fatigue life and strain energy for aged PVC/ENR TPEs as a result of TOA. At all sulfur loading fatigue life generally decreases after TOA. This may be due to the post curing phenomenon which enhanced the stiffness of the TPEs. The strain energy exponent $n$ also increases as a result of TOA. Figure 4 shows that the thermo-oxidative process has increased the sensitivity of the aged samples towards changes in the strain energy as compared to the unaged counterparts. This might be related to further cross-links formation during the TOA process. The increase in cross-link density as evidenced from the swelling index of the aged specimen (shown in Tab. 2) has equally influenced the rebound resilience (Fig. 2), which is lowered as the materials become stiffer due to additional cross-linking. Thus, it is expected that the aged samples possess higher $n$ values than the unaged counterparts, hence they exhibited higher hysteresis. This combined with the possibility of higher heat build up resulted in the lower fatigue life of the aged samples as compared to that of unaged counterparts.

## CONCLUSIONS

The incorporation of semi-EV sulfur vulcanizing system *via* dynamic vulcanization has improved the fatigue life of the plasticized PVC/ENR TPEs. As the sulfur concentration increases, the sensitivity of these materials towards strain energy decreases, hence, fatigue life increases. This has been attributed to the curative action which produced three dimensional network structure. The formation of cross-links as indicated from the swelling index data is expected to enhance the elasticity and the resilience of the rubber to a certain extent. This is believed to be the main factor which is responsible for the significant increase in the resistance of these materials against cyclic deformation. The thermo-oxidative aging process has adversely affected the fatigue life of the PVC/ENR TPEs. The increase in the cross-link density as indicated by the reduction in the swelling index data of the aged samples is associated with the post curing effect. The increased cross-link density stiffened and embrittled the materials. This has reduced both the elasticity and resilience of the cross-linked rubber particles in the samples. Consequently, the resistance of the aged PVC/ENR TPEs to cyclic deformation was reduced and shorter fatigue life were observed.

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