

# NOTES

## *Network Changes in V-Belt Rubber Base during Simulated Service Testing*

### INTRODUCTION

Attempts to improve the technical characteristics of rubber compounds for any product demand a thorough understanding of the mechanism of its failure. A sound knowledge about the network changes that are likely to occur in vulcanizates under service conditions will help in understanding their failure phenomena. Nando and De<sup>1,2</sup> have studied changes in the network structure of natural rubber (NR) vulcanizates subjected to different physical tests, since these tests, either singly or in combination, simulate at least a few service conditions. Stuckey and co-workers<sup>3</sup> have recently reported a decrease in the proportion of polysulfidic linkages in NR vulcanizates during De Mattia flexing test. Cunneen and Russel<sup>4</sup> reported changes in the network structure of NR tire tread vulcanizates during simulated and actual service testing. They found a marked reduction in the polysulfidic linkages and an increase in main chain modifications. An increase in the crosslink density in tire treads during service was observed by Howard and Wilder.<sup>5</sup> But the extent of the increase depends on the curative system and the nature of the base polymer. Similar studies using *cis*-1,4-polyisoprene have been reported by Podkolzina and co-workers.<sup>6</sup> Aging behavior of sealing materials under various environmental influences has been studied by Wolf.<sup>7</sup> However, more extensive studies in this line are required to understand the failure phenomena in detail. Mathew, Bhowmick, and De<sup>8</sup> have recently reported chemical and scanning electron microscopic (SEM) studies on the flexing fatigue failure of NR vulcanizates.

**V-Belts.** The popularity of rubber V-belts in the field of power transmission has been increasing in recent years, and this has resulted in extensive development work on belt design and the use of new materials. Use of stronger materials like rayon, steel wire, and polyester fibers as tension member demands proportionate improvements in the rubber components of transmission belts. The major technical requirements of the rubber components of V-belts are:

- (1) Good dynamic properties (high resilience, low heat buildup).
- (2) Good heat resistance.
- (3) Excellent resistance to flex cracking.

Moreover, these compounds should possess other desirable characteristics like resistance to oils and ozone.

Studies on the failure mechanism of V-belts are very scanty. The different ways in which V-belts fail have been outlined by Johnson and Hornung.<sup>9</sup> According to these authors, breakage of jacket, separation between cord and rubber or between cushion rubber and reinforced compression rubber, breakage of cord, and cracks in rubber resulting from alternating tension and compression are the main types of plane base belt failure. The influence of metal oxide dispersion on neoprene V-belt fatigue failure has been studied by Vickery, Fullman, and Snyder.<sup>10</sup> Walter<sup>11</sup> also reviewed the different types of failure in V-belts. However, more fundamental studies on the different aspects of failure are required to understand the total failure of V-belts. In the present work we have studied the chemical changes in the V-belt base rubber vulcanizates during simulated service testing. The effect of antioxidant on the failure process has been included in this study.

### EXPERIMENTAL

The formulations of the base mixes are given in Table I. We have chosen a blend of NR and polybutadiene rubber (BR) for these mixes. Mixing of the ingredients other than the curatives was carried out in a Shaw Intermix size K<sub>4</sub>. The mixing cycle is shown in Table II. The curatives were added on an open mill. The optimum cure time of the compounds are given in Table III. The mixes were used for the base of V-belts of size A 57. The tension member used was resorcinol-formaldehyde-latex (RFL)-treated rayon monocord. A natural-rubber-based compound, suitable for bonding to rayon cord, was used as the cushion rubber. The jacket used was from polychloroprene rubber based cotton fabric. The building of the belt was done on a collapsible drum, and the cut sections were jacketed and then cured in a steam pot for 30 min at 6.5-kg steam pressure.

TABLE I  
Formulations of the Mixes

| Mix no.                           | A    | B    |
|-----------------------------------|------|------|
| Natural rubber <sup>a</sup>       | 75   | 75   |
| Polybutadiene rubber <sup>b</sup> | 25   | 25   |
| Peptiser (Renacit VII)            | 0.1  | 0.1  |
| Zinc oxide                        | 10   | 10   |
| Stearic acid                      | 1.2  | 1.2  |
| Phenyl- $\beta$ -naphthylamine    | 1.25 | —    |
| Acetone-diphenylamine condensate  | 1.25 | —    |
| HAF black (N 330)                 | 75   | 75   |
| SRF black (N 770)                 | 25   | 25   |
| CI resin                          | 2.5  | 2.5  |
| Aromatic oil (Dutrex R)           | 10   | 10   |
| Retarder (Santoguard PVI-50)      | 0.43 | 0.46 |
| CBS                               | 0.68 | 0.68 |
| MBTS                              | 0.11 | 0.11 |
| Sulfur                            | 1.36 | 1.36 |

<sup>a</sup> Smoked sheets, RMAI.

<sup>b</sup> Cisamer 1220, as supplied by Indian Petrochemicals Corporation Ltd., Baroda.

TABLE II  
Mixing Cycle in Internal Mixer

| Time (min) | Charge       |
|------------|--------------|
| 0          | NR + BR      |
| 1          | Chemicals    |
| 2          | Carbon black |
| 3          | Oil          |
| 5.5        | Dump         |

TABLE III  
Physical Properties of the Vulcanizates<sup>a</sup>

| Properties  | A    | B    |
|---|------|------|
| 300% Modulus (MPa)  | 14.2 | 15.0 |
| Tensile strength (MPa)  | 16.9 | 17.1 |
| Elongation at break (%)   | 340  | 340  |
| Tear resistance (kN/m)  | 76.6 | 70.7 |
| Resilience (%)  | 29   | 38   |
| Hardness (Shore A)  | 74   | 75   |
| Heat buildup $\Delta T$ , ( $^{\circ}$ C)                           | 56   | 42   |
| Compression set <sup>b</sup> (%)                                    | 54   | 47   |
| Flexing resistance to failure (kcycles)                             | 125  | 65   |
| Abrasion loss <sup>c</sup> (cc/1000 rev)                            | 0.23 | 0.33 |
| Performance of the belt in the rig test <sup>d</sup> (h to failure) | 108  | 220  |

<sup>a</sup> Cured to optimum cure times at 150 $^{\circ}$ C (A, 7.5 min.; B, 7 min).

<sup>b</sup> ASTM Method B, 22 h, 25% compression, 70 $^{\circ}$ C.

<sup>c</sup> Obtained from Croydon-Akron abrader.

<sup>d</sup> Load, 65 kg; speed, 1400 rpm.

TABLE IV  
Chemical Characterization of the Vulcanizates<sup>a</sup>

| Sample description                                   | Mix no.            | Free sulfur<br>(mmol/kg RH) | ZnS sulfur<br>(mmol/kg RH) | Combined sulfur<br>[S <sub>c</sub> ] (mmol/kg RH) | V <sub>r</sub> | Polysulfidic<br>linkages (%) | Sol content<br>(mmol/kg RH) |
|--|--------------------|-----------------------------|----------------------------|---|----------------|------------------------------|-----------------------------|
| Optimum cured samples, prepared<br>in the laboratory | A                  | 73                          | 5                          | 347   | 0.211          | 26                           | 672                         |
|  | B                  | 68                          | 8                          | 349   | 0.227          | 29                           | 566                         |
| V-belt base, original                                | A                  | 15                          | 27                         | 383   | 0.225          | 24                           | 275                         |
|  | B                  | 27                          | 18                         | 379   | 0.248          | 30                           | 440                         |
| V-belt base, after rig<br>testing                    | A                  | 7                           | 29                         | 390   | 0.277          | 17                           | 347                         |
|  | Failed after 108 h | (53)                        | (7)                        | (1.8)   | (23)           | (29)                         | (26)                        |
|  | B                  | 20                          | 23                         | 383   | 0.288          | 16                           | 463                         |
|  | Run for 108 h      | (26)                        | (27)                       | (1.1)   | (16)           | (46)                         | (5)                         |
| Failed after 220 h                                   | B                  | 13                          | 28                         | 384   | 0.266          | 9                            | 609                         |
|  | Failed after 220 h | (52)                        | (56)                       | (1.3)   | (7)            | (70)                         | (38)                        |

<sup>a</sup> Figures given in parentheses are the % change after rig testing.

**Rig Testing.** The belts were rig tested up to failure. The test conditions are given in Table III. As the belt from mix A failed after 108 h; one belt from mix B was run under the same conditions for a period of 108 h to find out the changes occurring during the same period of testing in the two different base vulcanizates.

**Determination of Chemical Characteristics.** Estimation of free sulfur, zinc sulfide sulfur, sulfur combined,  $V_r$  (an indication of the concentration of chemical crosslinks), and polysulfidic linkages have been described earlier.<sup>8</sup> Sol content of the samples was determined according to the method described by Bristow.<sup>12</sup>

## RESULTS AND DISCUSSION

The physical properties of the base vulcanizates are given in Table III. The higher compression set, heat buildup, and tear resistance and the lower resilience and abrasion loss show that the vulcanizate A is having a lower crosslink density compared to the vulcanizate B. Similar observations have been made earlier.<sup>13</sup> This is confirmed by the lower  $V_r$  value of vulcanizate A (Table IV). It is also seen from Table III that the test life of the belt made from Mix A, in rig testing, is only half of that of the belt made from mix B. Although this is quite unexpected, we believe that the antioxidants have affected the state of cure of the vulcanizate.<sup>14</sup> The lower state of cure leads to higher heat buildup during rig testing, which, in turn, might have contributed to bond failure between the jacket and rubber and then between cord and rubber and ultimately to failure of the belt.

The chemical characterization of the optimum time cured vulcanizates and that of the base vulcanizates are given in Table IV. The lower values of free sulfur and higher values of combined sulfur and  $V_r$  of the base vulcanizates show that the belts are over cured compared to the test specimens cured in the laboratory.

**Results of Rig Testing.** Although the percentage change in the concentration of free sulfur and ZnS sulfur are quite considerable, the actual quantities involved are very small and hence not significant. This is also confirmed by the constant value of combined sulfur [ $S_c$ ]. The major changes in the chemical characteristics of the vulcanizates include an increase in  $V_r$  and in sol content and a decrease in the polysulfidic linkages. The changes in  $V_r$  can be attributed to the occurrence of postcuring and crosslink destruction reactions.<sup>15</sup> Both these reactions are activated by the heat generated during the testing. In the case of base A, the postcuring reactions are more predominant, and hence the percentage increase in  $V_r$  is more. In the case of base B, the  $V_r$  value increases first and then decreases. This is due to the predominance of crosslink destruction over postcuring as the testing is continued. The concentration of polysulfidic linkages decreases with testing. The reduction is more in base B, which contains no antioxidant. The extent of chain scission occurring during the testing period is not remarkable. Also during the rig testing because of the presence of the jacket, the base rubber has no direct access to oxygen, and hence oxidative chain scission does not occur to any significant level.

## CONCLUSIONS

The V-belt made from the base compound containing antioxidant failed more quickly than the one without antioxidant, possibly due to the lower crosslink density in the former. The lower crosslink density causes higher heat buildup, leading to bond failure and ultimate failure of the belt. The crosslink density of the vulcanizate increases during the rig testing. Also there is a reduction in the concentration of polysulfidic crosslinks.

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