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Scanning Electron Microscopy Examination of the Fracture Surface of NR/SBR and NR/BR Blends

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INTRODUCTION

Blending of two or more rubbers is carried out for three main reasons,¹ (1) improvement in technical properties (2) better processing (3) lower compound cost. Many products in the rubber industry are based on blends in all or part of their construction. Tyres are typical examples of products in large-scale volume production. Natural rubber (NR)/styrene-butadiene rubber (SBR) and natural rubber/polybutadiene rubber (BR) blends are extensively used in the manufacture of tyres.² Scanning electron microscopy (SEM) is now widely employed to observe the microstructure of fracture surfaces and hence to study the fracture mechanisms.^{3–5} Fracture may be caused due to faults in the material or operating conditions. The fracture surfaces of polymeric materials show many characteristic features which enable the destructive influence to be recognized. Since the mechanical behaviour of blended rubbers is very sensitive to the filler distribution and level,^{6–8} curing aspects,^{9–11} compatibility of the rubbers^{12–14} etc. an attempt has been made to

observe the microstructure of the fracture surface of test samples in tension and abrasion of NR/SBR and NR/BR blends to correlate it with the strength and type of failure of these materials.

EXPERIMENTAL

Preparation of test samples

The test samples were prepared from filled 50/50 blends of NR & SBR and of NR & BR. The formulations employed are shown in Tables I and II. The compounds were prepared on a laboratory mixing mill at near ambient temperature (30°C). The rubber blends were prepared initially and then the other additives were added in the usual order as per ASTM D3182 (1982). The cure curves of the compounds were taken on a Monsanto rheometer model R-100 at 150°C. Then the compounds were vulcanized upto their respective optimum cure times on a steam heated laboratory hydraulic press. Dumb-bell samples were punched out along the mill grain direction from the moulded sheets of 15 × 15 × 0.2 cm size. The test pieces of 2 × 2 × 1 cm size were directly moulded for abrasion testing. The tensile testing and the abrasion resistance testing of the samples were done as per ASTM D 412 (1980) and ASTM D394 respectively.

TABLE I
Formulations of the NR/SBR vulcanizates

Vulcanizate	1	2	3	4
NR ^a	50.0	50.0	50.0	50.0
SBR ^b	50.0	50.0	50.0	50.0
ZnO	5.0	5.0	5.0	5.0
Stearic acid	2.0	2.0	2.0	2.0
PBN ^c	1.0	1.0	1.0	1.0
HAF black (N330)	40.0	40.0	40.0	40.0
Aromatic oil	4.0	4.0	4.0	4.0
CBS ^d	0.8	0.8	0.6	1.4
Sulphur	1.8	3.0	2.0	2.0

^a $\bar{M}_w = 7.70 \times 10^5$; Mooney viscosity, ML(1 + 4) at 100°C, 85.3; ISNR 5 (Rubber Research Institute of India)

^b 23.5% styrene; Mooney viscosity, ML(1 + 4) at 100°C, 49.2

^c *N*-Phenyl- β -naphthylamine; Indian Explosives Limited

^d *N*-cyclohexyl 1-2-benzothiazyl sulphenamide; Indian Explosives Ltd.

TABLE II
Formulations of the NR/BR vulcanizates

Vulcanizate	5	6	7	8
NR	50.0	50.0	50.0	50.0
BR ^c	50.0	50.0	50.0	50.0
ZnO	5.0	5.0	5.0	5.0
Stearic acid	2.0	2.0	2.0	2.0
PBN	1.0	1.0	1.0	1.0
HAF black (N330)	40.0	40.0	40.0	40.0
Aromatic oil	4.0	4.0	4.0	4.0
CBS	0.7	0.7	0.6	1.2
Sulphur	1.8	3.0	2.2	2.2

^c 97% 1,4 (cis); Mooney viscosity, ML(1 + 4) at 100°C, 48.0

SEM observation of the fracture surfaces

The failed tensile test piece and the abraded surfaces were stored in a dessicator to avoid contamination from dust particles. Fracture surfaces were sputter coated with gold within 24 hours of testing. SEM observations were made using a Philips 500 model scanning electron microscope.

RESULTS AND DISCUSSION

The tensile fracture surfaces of NR/SBR vulcanizates 1, 2, 3 & 4 (Table I) are shown in Figures 1, 2, 3 & 4 respectively. Increase in either sulphur or accelerator in the compound increases the crosslink density of the matrix which results in enhanced strength.⁹⁻

¹¹ This could be observed from the SEM photographs by the progressive increase in the roughness of the fracture surfaces³ between Figures 1 & 2 and Figures 3 & 4. This shows that 50/50 NR/SBR blend behaves more or less similar to that of single rubbers, producing a regular pattern in the network structure with a change in the concentration of the curatives.

Figures 5, 6, 7 & 8 show the tensile fracture surfaces of 50/50 NR/BR vulcanizates 5, 6, 7 & 8 (Table II). These figures suggest that the behaviour of 50/50 NR/BR blend is also similar to that of the 50/50 NR/SBR blend. However, between Figures 7 & 8 there is a marked difference (in Figure 8 the tearline becomes broader with

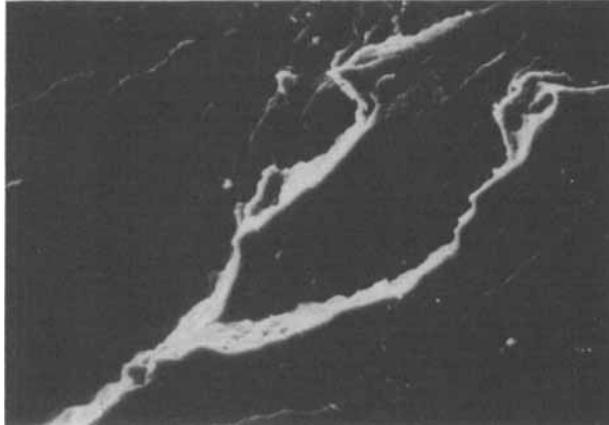


FIGURE 1

FIGURES 1-4 SEM photographs of the tensile fracture surfaces of 50/50 NR/SBR vulcanizates 1, 2, 3 & 4 ($\times 200$)

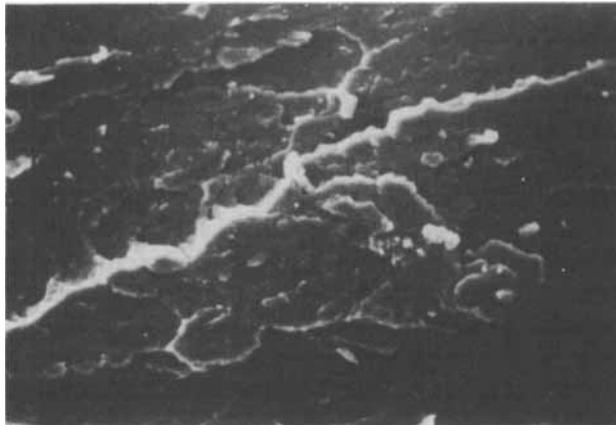


FIGURE 2

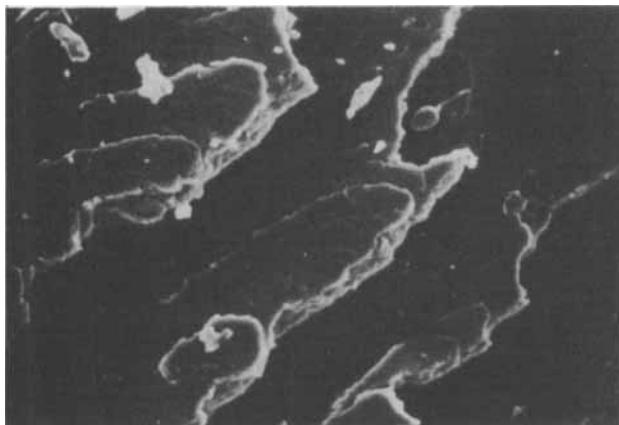


FIGURE 3

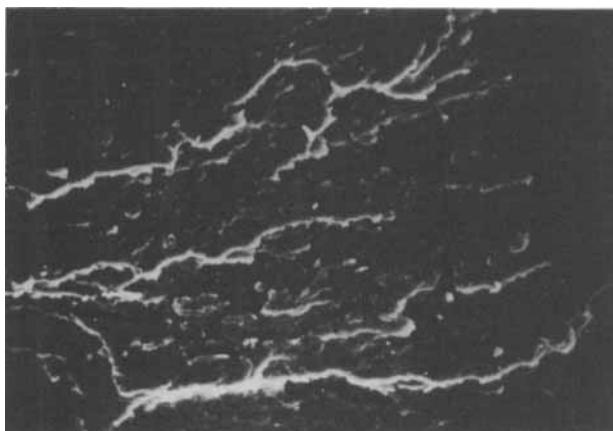


FIGURE 4

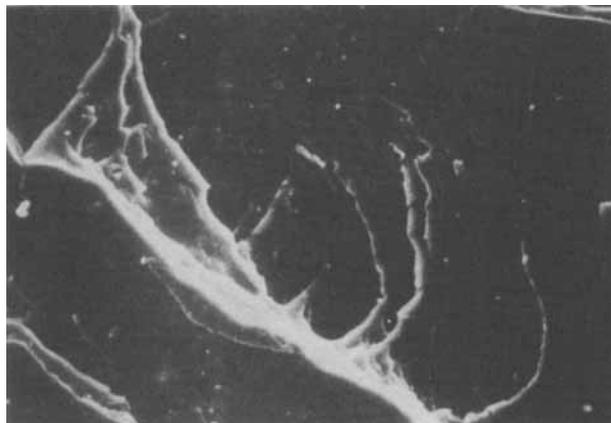


FIGURE 5

FIGURES 5-8 SEM photographs of the tensile fracture surfaces of 50/50 NR/BR vulcanizates 5, 6, 7 & 8 ($\times 200$)

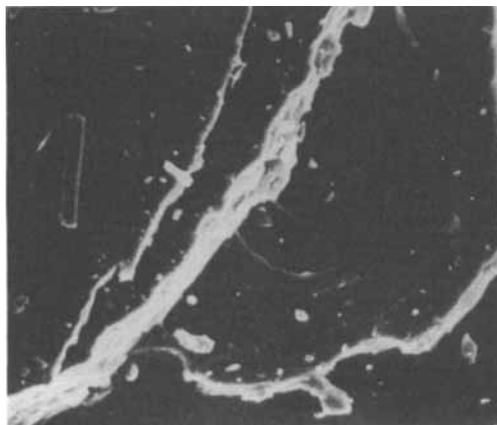


FIGURE 6

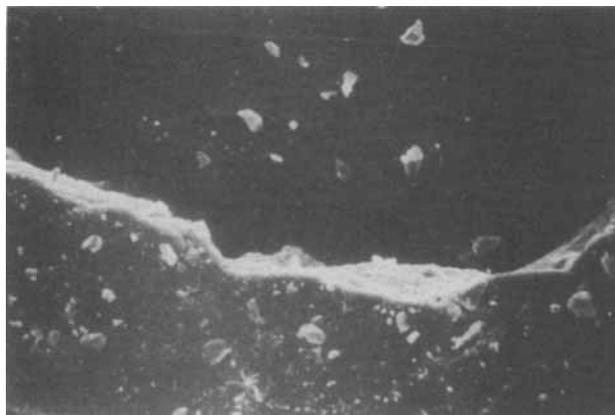


FIGURE 7

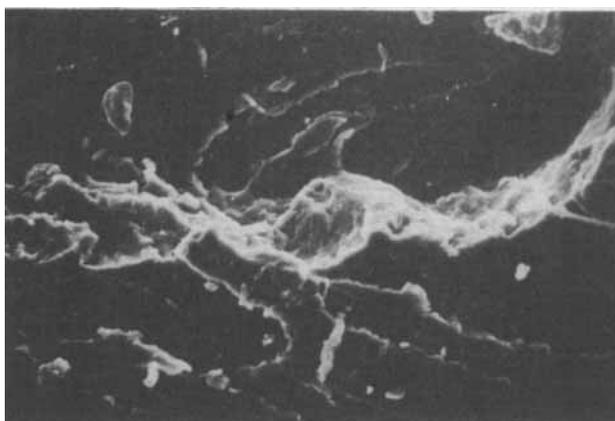


FIGURE 8

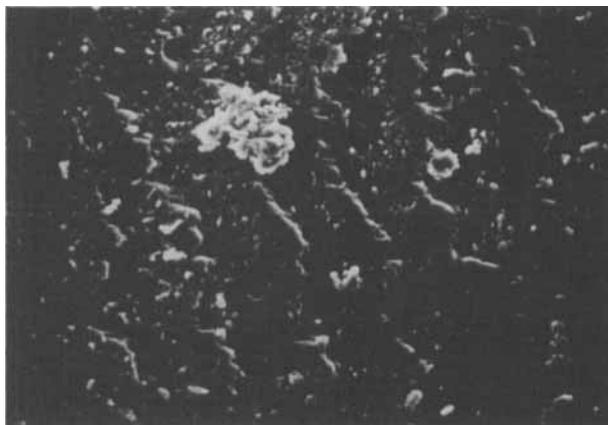


FIGURE 9

FIGURES 9-12 SEM photographs of the abraded surfaces of 50/50 NR/BR vulcanizates 5, 6, 7 & 8 ($\times 200$)

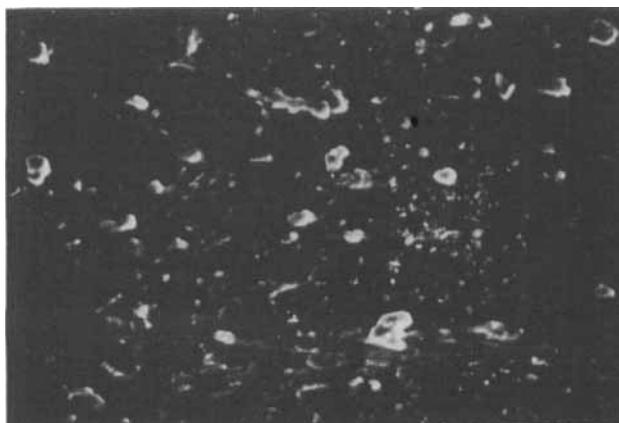


FIGURE 10

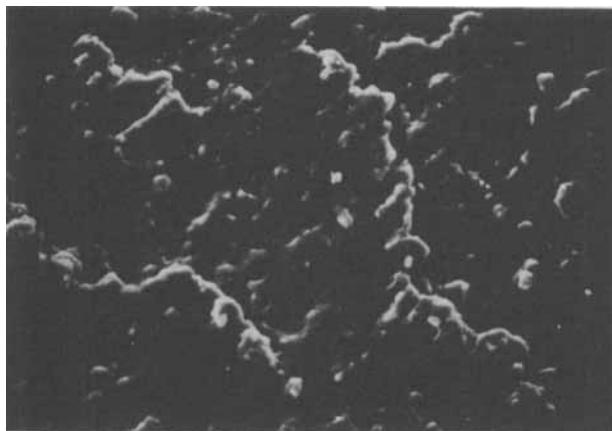


FIGURE 11

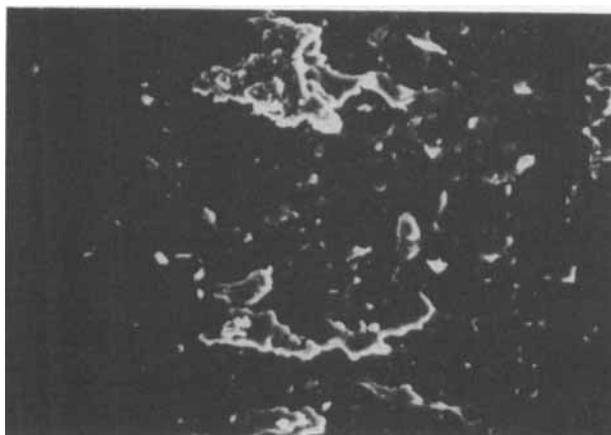


FIGURE 12

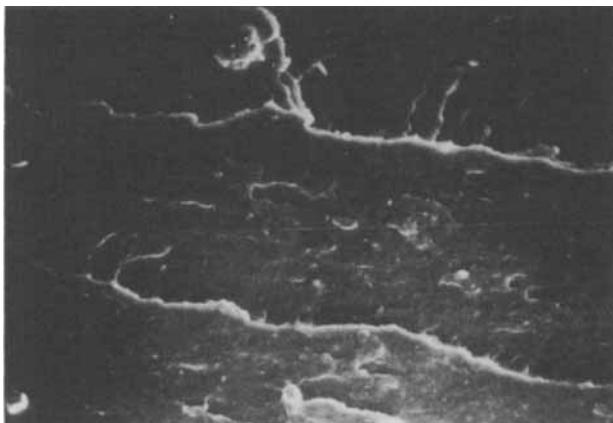


FIGURE 13

FIGURES 13–16 SEM photographs of the tensile fracture surfaces of aged 50/50 NR/BR vulcanizates 5, 6, 7 & 8 ($\times 200$).

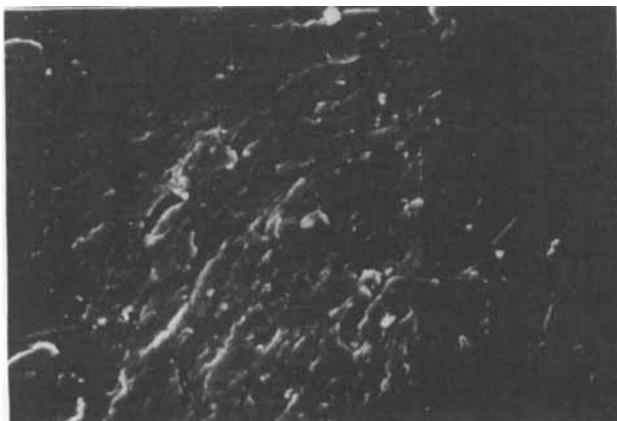


FIGURE 14

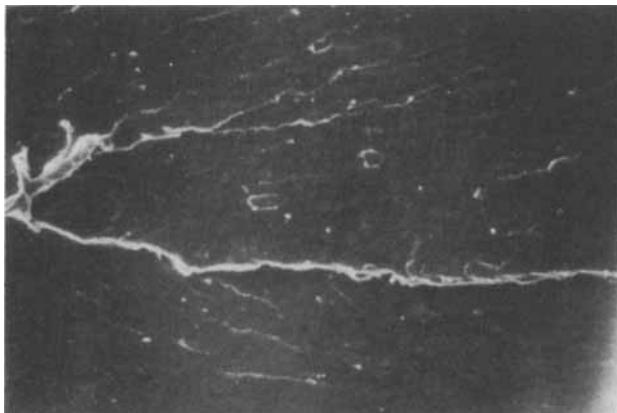


FIGURE 15

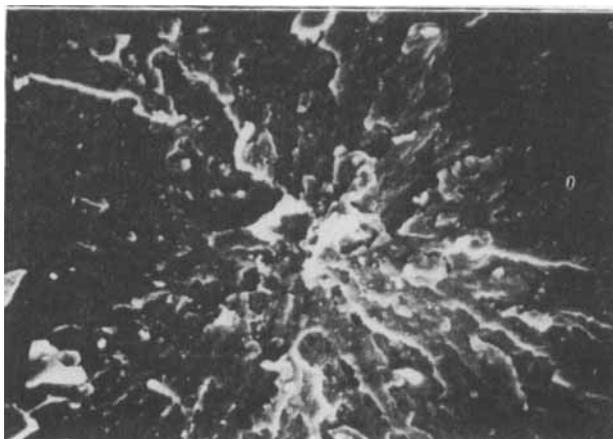


FIGURE 16

many branches) indicating that accelerator variation produces a greater change in the crosslink density and hence on the mechanical properties. Figures 9, 10, 11 & 12 show the abraded surface of 50/50 NR/BR vulcanizates 5, 6, 7 & 8 (Table II). In this case also a significant change is noticeable between Figures 11 & 12. Figure 11 shows a pattern indicating less resistance to abrasion whereas Figure 12 shows a much better abrasion resistance.¹³ This further shows that variation of accelerator, keeping the amount of sulphur constant produces a significant change in the network structure in the case of 50/50 NR/BR blend. Figures 13, 14, 15 & 16 show the tensile fracture surfaces of 50/50 NR/BR vulcanizates 5, 6, 7, 8 (Table II) after ageing. The pattern is more or less similar to those of the unaged samples. However, there is a significant increase in the roughness of the surfaces with ageing which indicates that the matrix becomes more brittle with ageing.

CONCLUSION

The study shows that variation of the amounts of sulphur or accelerator in 50/50 blends of NR/SBR and NR/BR produces effects similar to those produced in NR, SBR or BR and hence designing suitable formulations for attaining required vulcanizate properties for these blends could be done as in the case of the corresponding single rubbers. The study further shows that scanning electron microscopy studies of the fracture surfaces could be valuably used to assess the fracture mechanisms and physical properties of rubber blends.

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