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Effect of varied carbon black distribution on the morphology and properties of blends of natural and nitrile rubber

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Abstract

Morphology and properties of blends of 40/60 natural rubber (NR)/ high nitrile rubber (NBR, 45% acrylonitrile) were investigated as a function of distribution of carbon black between the two rubber phases. Carbon black distribution in the blends was varied by varying the level of carbon black in each rubber phase using a two-stage mixing technique. Results from cure testing indicated that cure characteristics are not sensitive to varied carbon black distribution. The morphology of all three blends containing either 10%, 50% or 90% of the carbon black initially in each elastomer, analysed with a transmitted light microscope was co-continuous. Morphology of the blend containing 90% of carbon black in the NBR phase was coarse textured. The blend containing 90% of carbon black in the NR phase had the smallest domains which were found to be the most elongated. The observed difference in morphology between these blends was attributed to the variation in the viscosity difference between the pairs of masterbatches combined in these blends. The smaller the difference in viscosity between the two masterbatches, the finer and more elongated the morphology. Modulus, hardness and tear strength increase with a higher content of carbon black in the NBR phase, whereas tensile strength, abrasion resistance and oil/fuel resistance increase with a higher content of carbon black in the NR phase. Compression set is lowest with carbon black equally distributed between the two phases.

Key words: carbon black distribution, mechanical blending, natural rubber, nitrile rubber, rubber – rubber blends

Introduction

Rubber-rubber blends can be prepared by several methods namely, latex blending, solution blending, latex and solution blending, mechanical blending and blending, powdered rubbers. Mechanical blending is the most widely used method and is the method employed in the preparation of 40/60 NR/NBR blends throughout this study. The above mentioned blending methods are subdivided into the two general categories namely, "pre-blending" and "phase mixing". Phase mixing is done by the preparation of individual masterbatches and subsequent blending of the same,

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mechanically. The masterbatch technique has found general acceptance in mechanical blending technology (Shundo *et al.*, 1966).

In general, physical properties of vulcanised rubber blend compounds are influenced by morphology and the distribution of compounding ingredients (especially reinforcing fillers like carbon black) between the phases of the blend. Carbon black distribution in rubber blends is governed by a combination of complex factors such as the ability of a rubber to wet and form strong attachments to the filler surface, relative mixing viscosities of the components, type of carbon black and the technique of blending. Effect of carbon black phase distributional variations on physical properties of blends of NR with synthetic rubbers such as polybutadiene (BR) and styrene-butadiene rubber (SBR) has been studied in the past. Hess *et al.* (1967) reported that abrasion resistance and tensile strength of 50/50 NR/BR blends were highest with most of the carbon black (N 220) in the BR phase (based on solution-mixed masterbatches). Tear strength was optimised with about 60% of the carbon black in the BR phase. Sircar *et al.* (1974) also reported low tensile strength for NR/BR and NR/SBR blends with a lower carbon black loading in the synthetic rubber. Krakowski & Tinker (1990) have also shown that preferential placement of the carbon black (N 330) in the BR phase of NR/BR blends significantly improves the abrasion resistance. However, studies of Hess and Chirico (1977) on 50/50 blends of NR/SBR and NR/BR showed only slight variations in modulus and tensile strength with different carbon black (N 220) phase distributions. Results of abrasion resistance also showed only a minimal dependence on location of carbon black (based on mechanically - mixed masterbatches). As in the case of the work of Hess *et al.* (1967) tear strength results showed significant variations as a function of carbon black location. In NR/SBR, the highest tear strength was achieved with most of the carbon black in the SBR phase. In contrast, the tear strength of the NR/BR was highest with most of the carbon black in the NR phase. In both cases, high tear strength was apparently related to a high carbon black loading in the continuous phase.

This research is focused on blends of NR and NBR prepared according to the masterbatch blending technique. The calculated solubility parameters of NR (δ_{NR}) and high nitrile rubber, 45% ACN (δ_{NBR}) are 17.19×10^3 $(Jm^{-3})^{1/2}$ and 20.46×10^3 $(Jm^{-3})^{1/2}$, respectively (Edirisinghe, 1999). Since the difference between these two values is three times the normal limit of miscibility ($10^3(Jm^{-3})^{1/2}$) (Bohn, 1968), the two rubbers are highly immiscible at molecular level. Properties of these immiscible NR/NBR blends are expected to vary with varied distribution of compounding ingredients, especially carbon black between the phases of NR/NBR blends as in the case of the other types of NR/synthetic rubber blends mentioned earlier.

The work reported in this paper describes the effect of varied carbon black distribution on rheological and physical properties of 40/60 NR/NBR (45% ACN) blends. Also, the paper discusses the rheological behaviour of NR and NBR masterbatches containing different amounts of carbon black.

Materials and methods

Materials

NR (SMR 10CV) was obtained from the MRPRA, UK and NBR, 45% ACN (Nipol DN001 W 45) was supplied by Zeon, UK. Carbon black (N 660) and sulphur were supplied by Cabot Carbon, UK and Schill & Seilacher, UK, respectively. The accelerators TMTD, CBS and TBBS together with PVI were obtained from Flexysys, UK. Zinc oxide and stearic acid were supplied by BDTP, UK.

Experimental

Experimental design

Mixing was conducted in two stages. The initial location and proportion of the carbon black in each rubber phase of the blend was varied by producing individual rubber/carbon black masterbatches in the first stage of the mixing cycle (Tables 1 and 2).

In the second stage, blending of the masterbatches followed by the addition of other compounding ingredients was carried out to produce 40/60 NR/NBR blends with either 10%, 50% or 90% of the carbon black located initially in each elastomer (Tables 3 and 4).

Table 1. *Stage 1: Masterbatch formulations, phr*

	A	B	C	D	E	F
NR	100	100	100	-	-	-
NBR	-	-	-	100	100	100
Carbon black	5	25	45	3.33	16.7	30

Table 2. *Stage1: Mixing sequence and the corresponding mixing times of the masterbatches*

	No. of working rotor revolutions	Mixing time at 60 rpm (s)
NR or NBR addition	0	0
Carbon black addition	90	90
Dump	210	210

Table 3. *Stage II : Masterbatch blend formulations*

	NR10NBR90 compound	**NR50NBR50 compound**	**NR90NBR10 compound**
Blend components	NR:NBR	NR:NBR	NR:NBR
Component ratio	40:60	40:60	40:60
% carbon black in NR	10	50	90
% carbon black in NBR	90	50	10
Carbon black loading, phr	20	20	20
Composition [a]			
MB.A	42	-	-
MB.B	-	50	-
MB.C	-	-	58
MB.D	-	-	62
MB.E	-	70	-
MB.F	78	-	-
ZnO	5	5	5
Stearic acid	0.5	0.5	0.5
Flectol H	2	2	2
TMTD	2	2	2
CBS	1	1	1
Sulphur	1	1	1
PVI	2	2	2

[a] MB = Masterbatch, see Table 1

Table 4. *Stage II: Mixing sequence and the corresponding mixing times of the masterbatch blends*

	No. of working rotor revolutions	**Mixing time at 60 rpm (s)**
NRMB + NBRMB + ZnO + stearic acid + Flectol H addition	0	0
TMTD + CBS + sulphur + PVI addition	60	60
Dump	120	120

Mixing was carried out at a rotor speed of 60 rpm using a computerized Francis Shaw K1 Intermix consisting of intermeshing rotors. A laboratory two-roll mill (J.R. Dare Ltd.) was used to sheet out each rubber mix discharged from the K1 Intermix by a single pass, while hot.

Evaluation of cure characteristics of the three masterbatch blends was carried out using the Wallace Shawbury Precision Cure Analyser, a rotorless instrument which deforms the test sample by the oscillatory motion of the lower half of the die cavity.

Rheological analysis

Rheological characterisation of the masterbatches and the masterbatch blend compounds was carried out using the Negretti TMS biconical rotor rheometer, which gives approximately uniform shear throughout the test specimen.

The relationship between shear stress and shear rate under conditions of steady state viscous flow was obtained between shear rates of 0.1 to $100 s^{-1}$ at the standard test temperature ($100^{\circ}C$). The shear rates applied in the ascending order and the corresponding step durations used, to achieve the steady - state are given in Table 5.

Shear stress values obtained directly from the microprocessor were plotted against time to ascertain the steady-state shear stress values corresponding to the seven shear rates employed. The results are presented in the form of logarithmic plots of apparent viscosity ($\eta_a = \tau / \square^{\cdot}$) against shear rate.

Analysis of morphology and carbon black distribution using microscopy

Thin sections (1 micron thick) of all the three vulcanised blends were prepared using a sledge type microtome fitted with a carbon dioxide cooling stage. Sections were then examined in a transmitted light microscope under bright field illumination. The phase structures of all three blends were photographed as video prints.

Table 5. *Testing sequence of shear rates and the corresponding step durations*

Step No.	Step duration (s)	Shear rate (s^{-1})	Sampling mode
1	35	0.1	Continuous
2	10	0.4	Continuous
3	10	1	Continuous
4	10	4	Continuous
5	5	10	Continuous
6	5	40	Continuous
7	5	100	Continuous

Physical testing

Physical testing of all the vulcanised blends was carried out as per the relevant British Standard. The tensile properties and tear strength were determined using a "Hounsfield 500L" tensile testing machine fitted with a laser extensometer (BS 903: Part A2 (1971)).

The abrasion resistance index (ARI) and the percentage compression set at room temperature (20°C) as well as at elevated temperature (70°C) were determined using the DIN abrader (BS 903: Part A9 (1988) - method A) and the compression set apparatus (BS 903: Part A6 (1969) - method B), respectively. The hardness was measured using a Shore A Micro Hardness Tester.

Swelling measurements

Swelling measurements of the three vulcanised blends were conducted at room temperature using toluene and ASTM oil No.2. Percentage increase in mass was calculated according to the equation given in ASTM: D 471 - 479.

Results and discussion

Rheology of NR and NBR masterbatches

Variation of apparent viscosity (η_a) of the three NR masterbatches containing 5, 25 and 45 phr of carbon black with shear rate is shown in Fig. 1.

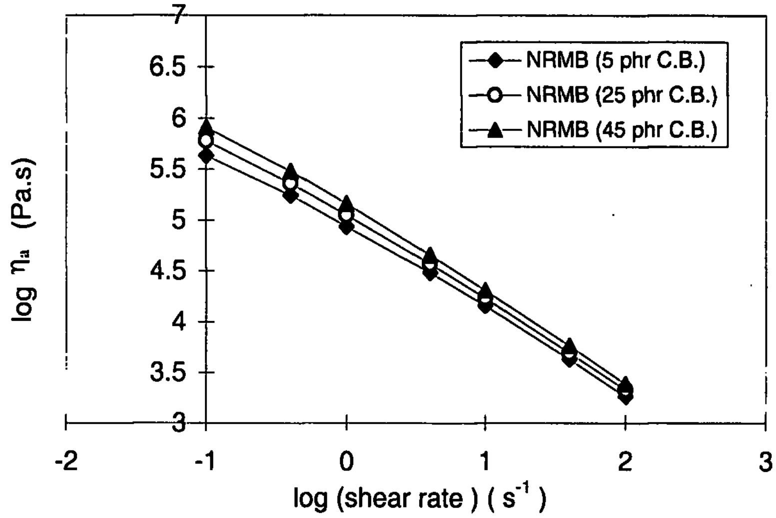

Fig. 1. Apparent viscosity against shear rate plots of NR masterbatches

It could be seen from the above plot that η_a of the masterbatches decreases in the order of NRMB (45 phr, carbon black), NRMB (25 phr, carbon black), NRMB (5 phr, carbon black) and this order is maintained throughout the shear rate studied. Thus the data reflect a rise in the viscosity with increasing proportion of carbon black. This is in accordance with the original Einstein equation as well as with the expanded version of the Einstein equation proposed by Guth and Gold, which applies to suspensions of non-attracting spherical particles (Walters & Keyte, 1962).

The difference in η_a between the three masterbatches is somewhat greater at lower shear rates and becomes smaller as the shear rate is increased. This implies that the influence of the proportion of carbon black on the viscosity diminishes at high shear rates. Kraus (1972), attributed this phenomenon to secondary aggregation effects which become smaller at high shear rates.

The apparent viscosity of the three NBR masterbatches containing 3.33, 16.7 and 30 phr of carbon black plotted against shear rate is shown in Fig. 2.

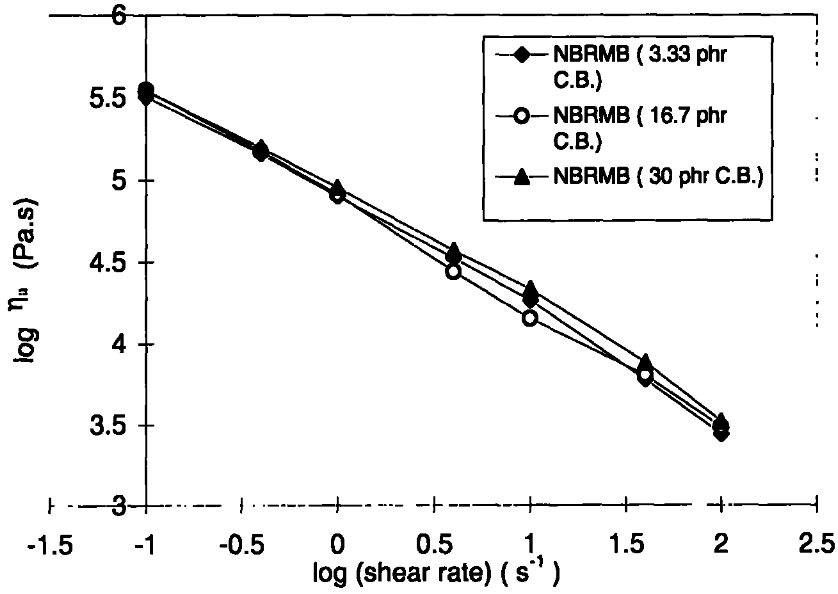

Fig. 2. Apparent viscosity against shear rate plots of NBR masterbatches

Unlike in the case of NR, the proportion of carbon black does not have a significant effect on the viscosity of NBR even at very low shear rates. In the case of NR, at low shear rates, increasing the amount of carbon black increases the viscosity as the stresses available for disagglomeration or in other words disintegration of agglomerates into aggregates are low. In the case of NBR, although the stresses available for disagglomeration are low at low shear rates, weak van der Waals' forces between aggregates, responsible for agglomeration may diminish due to strong interactions which may arise between the polar groups present on the carbon black surface and the polar – C≡N groups in NBR. Hence, the viscosity of NBR will not increase significantly with the loading of carbon black, unlike in the case of NR.

Apparent viscosity against shear rate plots of the pairs of masterbatches combined in the blends are given in Figures 3a, 3b and 3c. It is apparent from all the three figures that the drop in apparent viscosity of the NR masterbatch as the shear rate is increased is greater than that of the respective NBR masterbatch. It is also observed from the three figures that at high shear rates (>40 s^{-1}) corresponding to high rotor speeds in the mixer, the difference in viscosity between the pairs of masterbatches decreases in the order Fig. 3a, Fig. 3b and Fig. 3c. Thus the domain size of the blends would be expected to decrease in the order NR10NBR90, NR50NBR50, NR90NBR10.

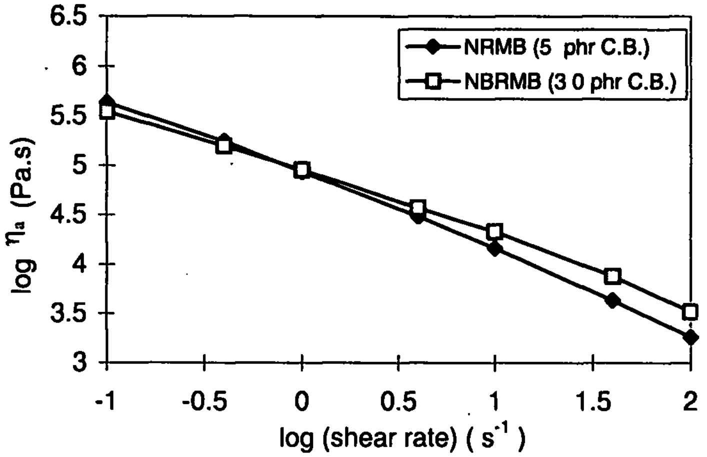

Fig. 3a. Apparent viscosity against shear rate plots of the pair of masterbatches combined in the NR10NBR90 blend

Effect of varied carbon black distribution

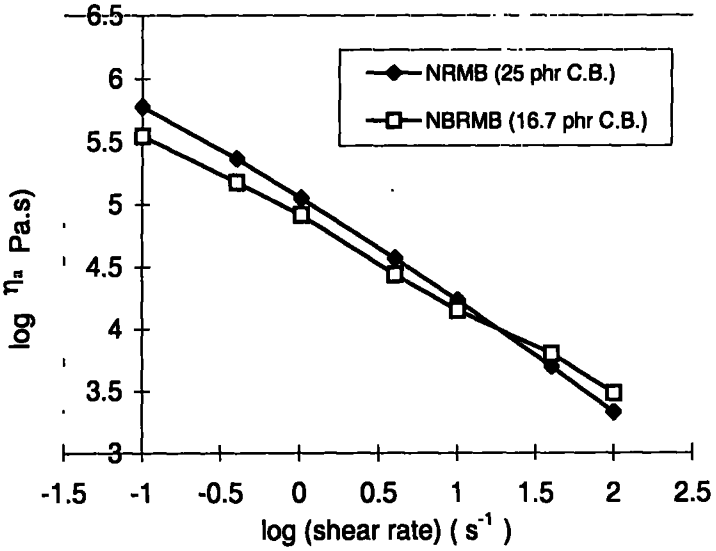

Fig. 3b. Apparent viscosity against shear rate plots of the pair of masterbatches combined in the NR50NBR50 blend

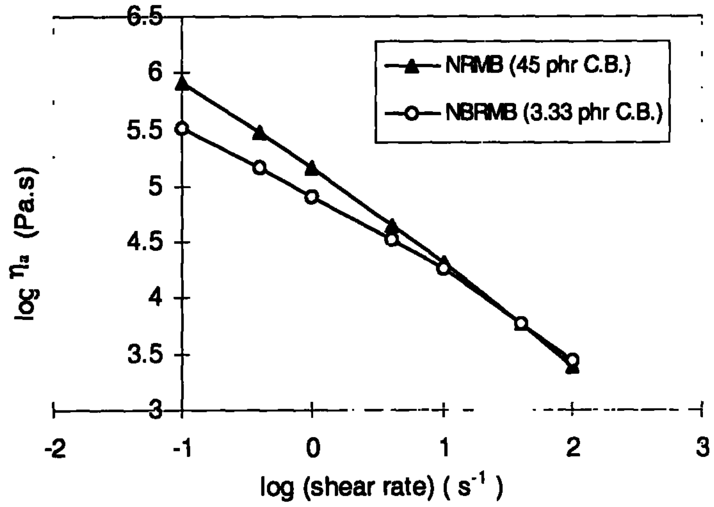

Fig. 3c. Apparent viscosity against shear rate plots of the pair of masterbatches combined in the NR90NBR10 blend

Effect of location of carbon black on the morphology of NR/NBR masterbatch blends

Video prints of the transmitted light micrographs of NR10NBR90, NR50NBR50 and NR90NBR10 blends at a magnification of 400X are shown in Fig. 4. Micrographs 4a, 4b and 4c clearly indicate that the contrast between the phases is predominantly due to the carbon black concentration as reported by Marsh *et al.* (1967). All three micrographs show a lighter and a darker phase. In the case of the NR50NBR50 blend the domains are diffused and cloudy in appearance. This is almost certainly due to the presence of a nearly equivalent loading of carbon black in the two phases. This observation suggests that no significant amount of carbon black migrates between the phases upon blending of masterbatches as reported by many researchers (Callan *et al.*, 1971, Lee & Singleton, 1979, Lee, 1981, Sircar & Lamond, 1973, Sircar *et al.*, 1974).

It is likely that the darker phase in micrograph (b) is NR and the lighter phase is NBR as the concentration of carbon black in the NR phase (25%) is higher than that of NBR (16.7%). It is more certain that the darker phase in micrograph (c) is NR, whereas in micrograph (a) it is NBR. It is also observed from the three micrographs that the morphology of all three masterbatch blends is towards co-continuous. The mean widths of the domains (lighter phase) of the NR10NBR90, NR50NBR50 and NR90NBR10 blends are approximately 7.5, 5 and 4µm, respectively. Therefore, the NR10NBR90 blend appears to be more heterogeneous than the other two masterbatch blends.

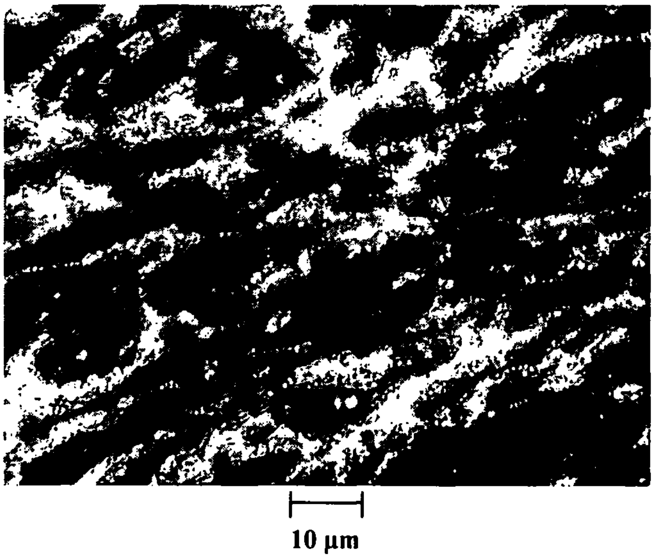

Fig. 4a. Transmitted light micrograph of the NR10NBR90 masterbatch blend at 400X magnification

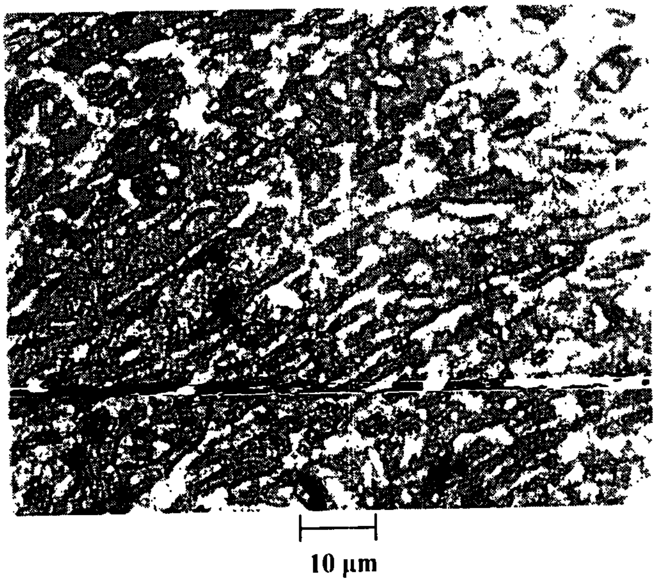

Fig. 4b. Transmitted light micrograph of the NR50NBR50 masterbatch blend at 400X magnification

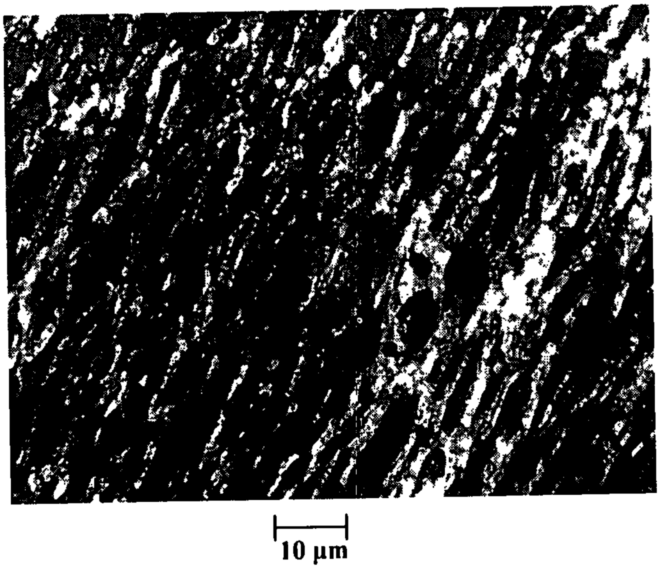

Fig. 4c. Transmitted light micrograph of the NR90NBR10 masterbatch blend at 400X magnification

The difference in apparent viscosity (log scale) of the pairs of masterbatches combined in the NR10NBR90, NR50NBR50 and NR90NBR10 blends at 40 s^{-1} shear rate (corresponding to high mixer rotor speed) is 0.248, 0.106 and 0.01 Pa.s, respectively. Hence, as mentioned earlier the difference in viscosity at high shear rates is greatest between the pairs of masterbatches combined in the NR10NBR90 blend, and this may account for its greater heterogeneity in relation to the other two masterbatch blends.

This morphological study indicates that a greater percentage of carbon black in the NR phase produces more intimate blending at high shear rates in comparison to that containing a greater percentage of carbon black in the NBR phase, solely due to the smaller difference in viscosity between the component masterbatches.

Effect of location of carbon black on the apparent viscosity of NR/NBR masterbatch blends

The predicted as well as the measured apparent viscosity values of the masterbatch blends plotted against the percentage of carbon black in the NR phase is shown in Fig. 5.

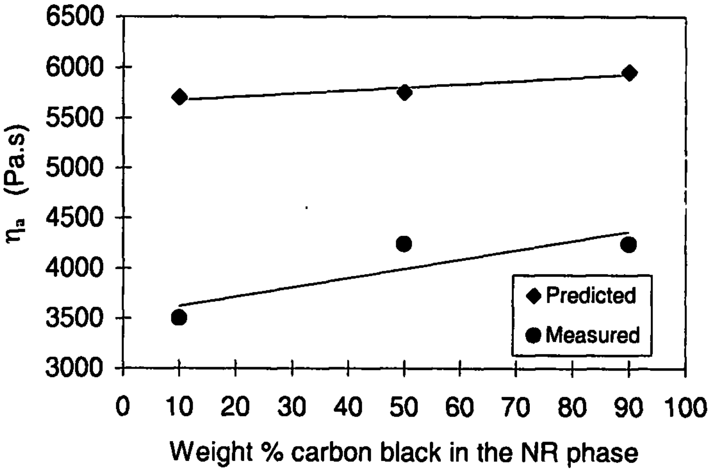

Fig. 5. Effect of location of carbon black on the apparent viscosity of NR/NBR masterbatch blends at 40 s^{-1} shear rate and 100°C

It can be seen from this figure that the predicted and measured trends in the variation of apparent viscosity with percentage of carbon black in the NR phase are more or less similar. The apparent viscosity of masterbatch blends increases slightly with more carbon black premixed into the NR phase. This is probably a reflection of the greater effect of carbon black on the apparent viscosity of NR in relation to NBR, as discussed earlier. In other words the location of carbon black in the individual rubber phases has a little effect on the apparent viscosity of NR/NBR blends. This is in agreement with the work of Lee (1981), based on shear viscosity measurements of SBR/BR blends.

Effect of location of carbon black on the cure characteristics of NR/NBR masterbatch blends

The scorch time, 90% cure time, cure rate index and (Max. - Min.) torque of the three NR/NBR masterbatch blends are given in Table 6.

No significant difference is observed between the cure characteristics of these masterbatch blends, despite the difference in morphology. This implies that the location of carbon black within the blended rubber phases does not significantly influence the cure characteristics of masterbatch blends.

Effect of location of carbon black on the physical properties of NR/NBR masterbatch blends

Modulus

The variation in moduli of the three masterbatch blends is shown in Figures 6a and 6b. NBR (45% acrylonitrile) vulcanisates formulated with carbon black have a modulus which is higher than that of comparable NR vulcanisates at low strain (Edirisinghe, 1999). Therefore, a masterbatch blend containing 90% carbon black in the NBR phase should give a higher modulus than a blend containing 90% of carbon black in the NR phase at low strain as shown in Fig. 6a.

Table 6. *Cure characteristics of NR/NBR masterbatch blends*

	NR10NBR90 compound	**NR50NBR50 compound**	**NR90NBR10 compound**
Scorch time (min.)	3.9	3.9	3.8
90% cure time (min.)	9.9	9.6	9.8
Cure Rate Index	16.6	17.5	16.6
(Max.-Min.) torque (Nm)	1.49	1.50	1.52

It is apparent from Fig. 6b that 300% modulus increases with increasing the amount of carbon black in the NBR phase. Modulus at 300% elongation is influenced by strain crystallization. Unlike in the case of NR, which has the ability to exhibit reinforcement by strain crystallization, reinforcing fillers must be added to NBR in order to achieve good mechanical properties. Hence, the results of 300% modulus are in agreement with the recognized need for carbon black reinforcement to develop optimum modulus of NBR and with the ability of NR to exhibit an alternate mechanism of reinforcement by strain crystallisation.

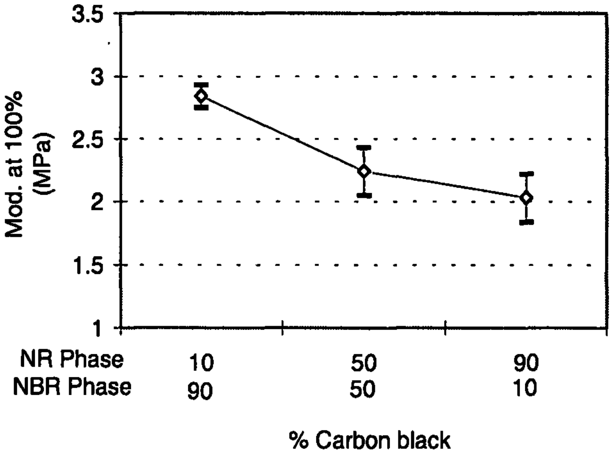

Fig. 6a. Influence of location of carbon black on the 100% modulus of masterbatch blends

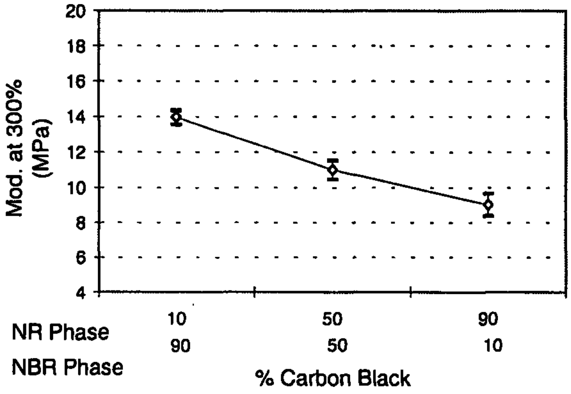

Fig. 6b. Influence of location of carbon black on the 300% modulus of masterbatch blends

Tensile strength

Fig. 7 shows the variation in tensile strength of the three masterbatch blends. The NR90NBR10 blend shows a higher tensile strength than the NR10NBR90 blend. However, there is no significant difference between the tensile strength values of NR90NBR10 and NR50NBR50 blends.

The significantly lower tensile strength obtained for the NR10NBR90 blend in comparison to the other two blends can be attributed mainly due to its coarse textured morphology. Thus for tensile strength morphology has a dominant effect over carbon black distribution. The trend in the variation of elongation at break is generally similar to that of tensile strength.

Tear strength

The tear strength of the NR10NBR90 blend is not significantly different from that of the NR50NBR50 blend, despite the large difference in morphology (Fig. 8).

This indicates that the tear strength of NR/NBR masterbatch blends is not significantly affected by morphology. Tear strength is highest with 90% of the carbon black in the NBR phase and lowest with 90% carbon black in the NR phase and the arguments put forward to explain the variation in modulus at 300% elongation can be treated as valid for tear strength.

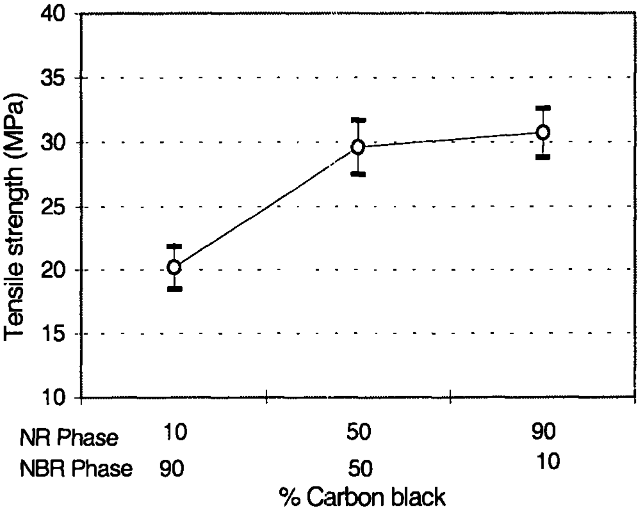

Fig. 7. Influence of location of carbon black on the tensile strength of masterbatch blends

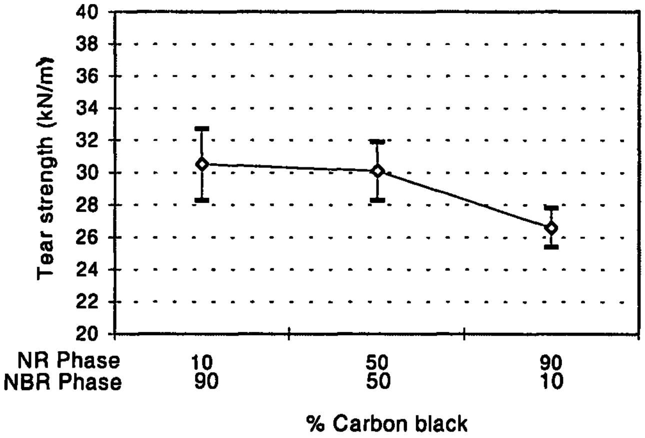

Fig. 8. Influence of location of carbon black on the tear strength of masterbatch blends

Hardness

As expected, the trend in the variation of hardness is similar to that of the modulus at 100% elongation. The high hardness of the NR10NBR90 blend (Fig. 9) could be due to the rigid structure inherited through the high-carbon black-NBR three dimensional mesh configuration (Fig. 4a). The results suggest that the hardness of masterbatch blends is affected by carbon black distribution via morphology.

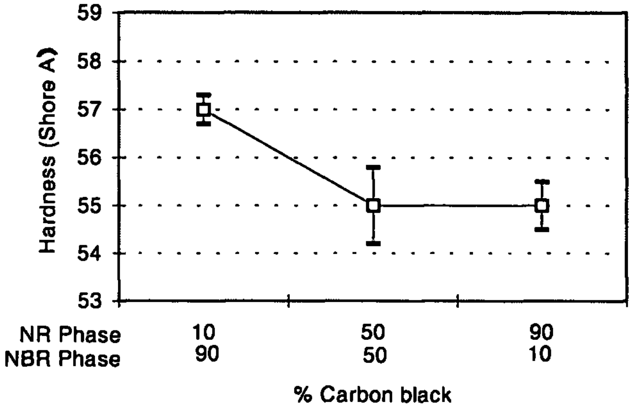

Fig. 9. Influence of location of carbon black on the hardness (Shore A) of masterbatch blends

Abrasion resistance

As in the case of tensile strength and elongation at break, the abrasion resistance is highest with most of the carbon black in the NR phase (Fig. 10).

Since the domain size of the NR10NBR90 blend is significantly larger than that of the NR50NBR50 and NR90NBR10 blends, the abrasion resistance of the former is expected to be higher than that of the latter blends (Edirisinghe, 1999). However, the abrasion resistance of the NR10NBR90 blend is markedly lower than that of the NR90NBR10 blend. This implies that the effect of location of carbon black on the abrasion resistance of masterbatch blends dominates the effect of morphology. NBR compounds containing reinforcing fillers are known to have an abrasion resistance which is about 30% greater than that of comparable NR compounds (Zhang, 1984). Therefore, a NR/NBR blend containing a low level *e.g.* 10% of carbon black (abrasion differences between the rubbers diminish with increasing the carbon black loading) in the NBR phase is expected to have a higher abrasion resistance than a blend containing the same amount of carbon black in the NR phase, which is in agreement with the results.

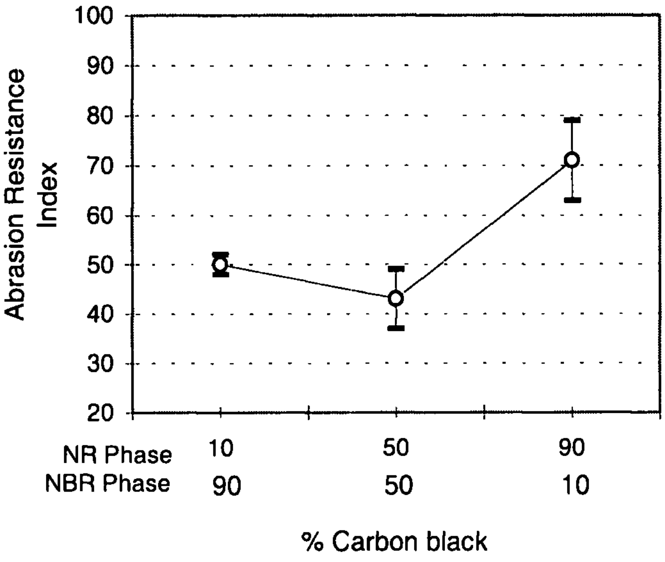

Fig. 10. Influence of location of carbon black on the abrasion resistance (DIN) of masterbatch blends

Compression set

Figures 11a and 11b show the variation in compression set of masterbatch blends obtained at room temperature and 70°C, respectively. The compression set of the NR50NBR50 blend is lower than that of the other two masterbatch blends at both the temperatures. The lower compression set shown by the NR50NBR50 blend suggests that the carbon black should be equally distributed between the phases in order to reduce compression set. This supports one of the requirements for minimizing compression set in single elastomer compounds proposed by Farid (1993).

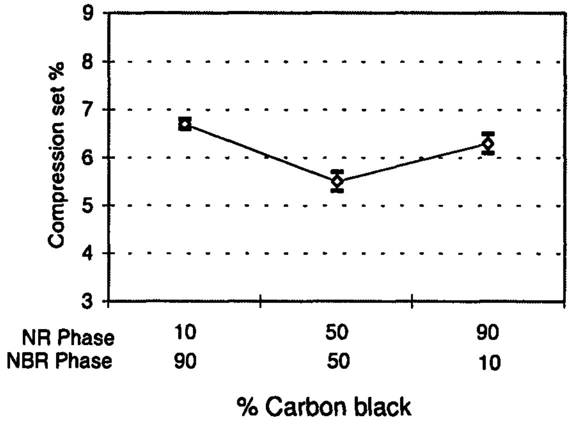

Fig. 11a. Influence of location of carbon black on the compression set (at 20°C) of masterbatch blends

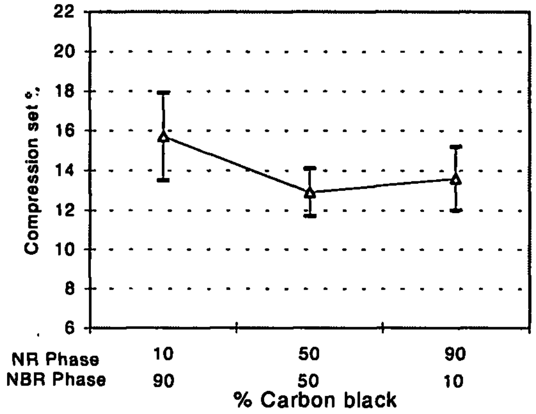

Fig. 11b. Influence of location of carbon black on the compression set (at 70°C) of masterbatch blends

Swelling measurements

Figures 12a and 12b show the variation in percentage swell of masterbatch blends in ASTM oil No. 2 and toluene, respectively.

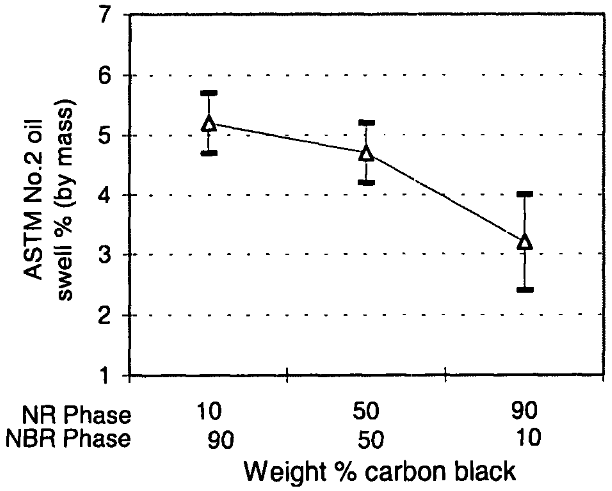

Fig..12a. Influence of location of carbon black on the percentage swell of masterbatch blends in ASTM oil No. 2

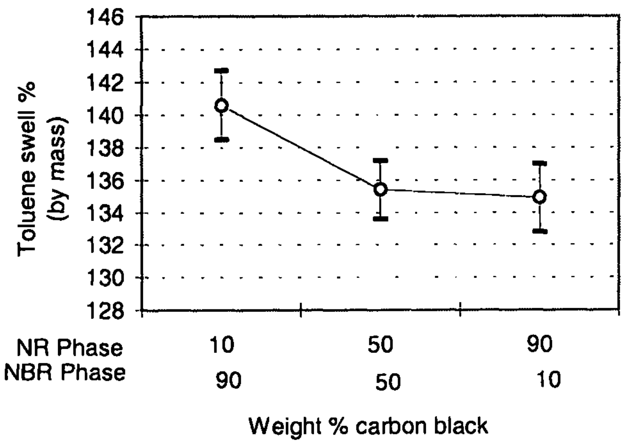

Fig. 12b. Influence of location of carbon black on the percentage swell of masterbatch blends in toluene

The oil uptake decreases with more carbon black premixed into the NR phase. Published ratings (Kairi Nagdi, 1993) indicate that NBR is far more resistant to ASTM oil No. 2 than NR. Lee (1985) has shown that the NBR phase of SBR/NBR masterbatch blends is almost impenetrable by ASTM oil No.2 and the penetrability is unaffected by carbon black loading. Thus the degree of swelling of NR/NBR masterbatch blends in ASTM oil No.2 should be governed by the degree of swelling of the NR phase. Thus the observed variation in oil swelling resistance between the masterbatch blends can be ascribed to the amount of carbon black present in the NR phase. Resistance to toluene of NBR is not much greater than that of NR (Jacques, 1971) and hence the NBR phase is also penetrable by toluene. Therefore, in toluene, the concentration of carbon black will affect the swelling of both NR and NBR phases. As a consequence, the carbon black distribution will not have such a great effect as in the case of the oil, where concentration of carbon black only affects swelling of the NR phase.

Conclusions

1. The location of carbon black in the individual rubber phases of NR/NBR masterbatch blends has a little effect on viscosity. Viscosity of the blend increases with more carbon black mixed into the NR phase.
2. Cure characteristics of NR/NBR masterbatch blends are not significantly affected by the location of carbon black in the individual rubber phases.
3. Moduli and tear strength of NR/NBR masterbatch blends is found to be highest with most of the carbon black in the NBR phase. The increase in 300% modulus and tear strength on increasing the amount of carbon black in the NBR phase is in agreement with the recognised need for carbon black reinforcement to develop optimum modulus and tear strength of NBR and the ability of NR to exhibit an alternate mechanism of reinforcement by strain crystallisation.
4. Abrasion resistance of NR/NBR masterbatch blends is found to be highest with most of the carbon black in the NR phase.
5. Percentage compression set of NR/NBR masterbatch blends at ambient as well as elevated temperature is lowest with carbon black equally distributed between the phases.
6. ASTM oil uptake of NR/NBR masterbatch blends decreases on increasing the amount of carbon black in the NR phase. The resistance of the NBR phase to swelling by ASTM oil No. 2 is unaffected by carbon black loading and hence the degree of swelling of these blends in ASTM oil No. 2 is governed by the degree of swelling of the NR phase, which decreases

on increasing the carbon black loading.

7. Toluene (or fuel) uptake also decreases on increasing the amount of carbon black in the NR phase, but to a lesser extent than with the oil. This is because the carbon black loading affects swelling of both NR and NBR phases, since toluene causes substantial swelling of both the elastomers.

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