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Aging and Mechanical Properties of NR/BR Blends

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The mechanical properties and post-thermal aging properties of natural rubber (NR) and polybutadiene rubber (BR) blends at different blending ratios are investigated herein. The experimental results show that both tensile and tear strengths of NR/BR blends increase with increasing NR content. BR has a higher compression stiffness than NR. The deformation of BR is less than that of NR under the same load conditions. With regard to aging properties, both tensile stress and strain of NR/BR blends decrease after prolonged aging. In addition, the stress loss of BR is lower than that of NR, meaning that the aging resistance property of BR is superior to that of NR. Furthermore, accumulated thermal history has shifted the glass transition temperature (T_g) of NR/BR blends toward lower temperatures while the loss tangent (tan δ) value increases with prolonged thermal aging.

Keywords compression stiffness, glass transition temperature, loss tangent, natural rubber, polybutadiene rubber

1. Introduction

The use of polymer blends is an effective method for altering the performance of polymer materials and is widely employed in engineering plastics, rubber, and fiber materials (Ref 1-13). Natural rubber is also commonly used, and it has certain advantages, such as flexibility. Nevertheless, some of its properties fall short in certain applications, such as oil resistance, air permeability, ozone resistance, compression set, and thermal aging resistance. Several studies have already dealt with the blends of NR and different rubber combinations. Chiu et al. (Ref 14-16) have explored the processibility, physical properties, and compatibility of NR/ENR and ENR/CR. Hamed et al. (Ref 17) studied the physical effects of NR/SBR after thermal aging. Sirisinha et al. (Ref 18) explored NR/NBR viscoelasticity and oil resistance, and Koshy et al. (Ref 19) studied the changes in NR/EVA physical and ozone resistance properties after thermal aging. In addition, there have been several reports on NR/BR blends (Ref 20-25) mainly dealing with the physical properties of NR/BR blends such as wear resistance, bending resistance, tensile strength, and compression stiffness. Several researchers have also explored the effect of carbon blacks or additives on NR/BR blends (Ref 26-28). Bristow et al. (Ref 29) analyzed the changes in properties on NR/BR blends after thermal aging. In addition, Mallik et al. (Ref 30) studied the thermal aging effect on the physical properties of NR/BR blends at different blending ratios. Using thermal analyses (thermogravimetric analysis [TG]; derivative thermogravimetric analysis [DTG]; differential thermal analysis [DTA]), Budrugeac (Ref 31) explored the reactions of heat and heat resistance in nonisothermal conditions. Meanwhile, Ciutacu et al. (Ref 32) used infrared (IR) spectrometry methods to investigate the molecular chain reactions of blends at higher temperatures. However, the mechanical properties, physical properties changes after aging, air permeability, and dynamic mechanical properties of NR/BR blends have rarely been studied. Such investigations will be useful for determining the basic properties of NR/BR blends, the influence of thermal aging, and the relationship with the molecular chain motion.

2. Experimental

2.1 Blending

Natural rubber (NR#3) and Taiwan Synthetic Rubber's polybutadiene rubber (BR1220) were mixed with vulcanizing agents, accelerators, and other materials to produce materials with the compositions shown in Table 1. At room temperature, NR, BR, and other additives were placed in a high-pressure Banbury kneader (SYD-5 model, Hsing Wang Co., Taiwan) for mastication. After 8 min of mastication, the material was mixed in a two-roller mixer (TMR-13, 8 in. diameter × 20 in. long, Gang Cheng Machinery Co.). The mixing roller to speed ratio was 1.2:1.0, and the roller distance was 10 mm, which was gradually shortened to 5 mm. After 30-40 rounds of thinning, an end product of green rubber material was obtained. The additive procedure is shown in Table 2.

2.2 Vulcanization

At a heating temperature of 140 °C, a heat presser was used for vulcanization of NR/BR blends with six different blending ratios and heating times, as shown in Table 3, producing specimens with thicknesses of 2 mm and 12.5 mm.

2.3 Physical-Mechanical Property Analysis

Specimens for testing tensile strength and elongation at breaking were cut in accordance with an ASTM-D412-92 C-type specimen, using a Universal Tensile Tester (Orientec Corp., Japan) at a tensile velocity of 500 mm/min. Test method for vulcanized rubber and thermoplastic rubbers and thermoplastic elastomers-tension (1996). For testing the tear strength, the specimens were cut and prepared in accordance with an ASTM-D624-91 C-type specimen, at a tensile velocity of 500 mm/min. Test method for tear strength of conventional vulcanized rubber and thermoplastic elastomer (1996).

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Table 1 Composition of NR/BR blends

Ingredients, phr	NR/BR ratio 0/100	20/80	40/60	60/40	80/20	100/0
BR1200	100	80	60	40	20	0
NR#3	0	20	40	60	80	100
Pine tar oil	7	7	7	7	7	7
MgO	4	4	4	4	4	4
ZnO	5	5	5	5	5	5
S_8	1.8	1.8	1.8	1.8	1.8	1.8
HAF-330	45	45	45	45	45	45
PPD	1.8	1.8	1.8	1.8	1.8	1.8
Wax	1.8	1.8	1.8	1.8	1.8	1.8
TMTD	0.1	0.1	0.1	0.1	0.1	0.1

Total: 166.5 phr

phr, part per hundred parts of rubber; BR1200, polybutadiene rubber; NR#3, natural rubber; pine tar oil, softener; MgO, magnesium oxide; ZnO, zinc oxide; S_8, sulfur; HAF-330, carbon black (N330); PPD, *N,N′*-dialkyl-*p*-phenylene-diamines; Wax, ozone resistor; TMTD, tetramethyl thiuram disulfide

Table 2 Additive procedure and mixing time for NR/BR blends

Additive procedure	Elapsed time, min	Mixing time, min
1. NR/BR	0~2	2
2. Formula mixing	2~5	3
3. Carbon black mixing	5~8	3

Table 3 Cure time for NR/BR blends at 140 °C

Sample thickness, mm	Cure time, min NR/BR 0/100	NR/BR 20/80	NR/BR 40/60	NR/BR 60/40	NR/BR 80/20	NR/BR 100/0
2	15	12	9	9	7	7
12.5	20	17	14	14	12	12

2.4 Compression Set of the Vulcanized Rubber

A circular specimen with a diameter of 29 ± 0.5 mm and thickness of 12.5 ± 0.5 mm was prepared in accordance with ASTM-D395-89 and tested at 70 °C for 22 h. Test method for rubber property-compression set (1996).

2.5 Compression Stiffness of the Vulcanized Rubber

A circular specimen is prepared with a diameter of 29 ± 0.5 mm and a thickness of 12.5 ± 0.5 mm. Its compression stiffness is tested using an MTS-810 TYPE Material Testing System (MTS Systems Corp.), under the condition of a 7 mm compression deformation. The experimental setup for compression stiffness is shown in Fig. 1.

2.6 Air Permeability of the Vulcanized Rubber

A circular, vulcanized rubber specimen 2 mm thick and with a diameter of 90 mm was prepared. The permeable gas flow was measured through the specimen, at room temperature, by means of a Toyoseiki-GTR gas permeability analyzer (Taiwan). The volume flow rate of the gas permeating through the specimen was measured by the flow algorithm.

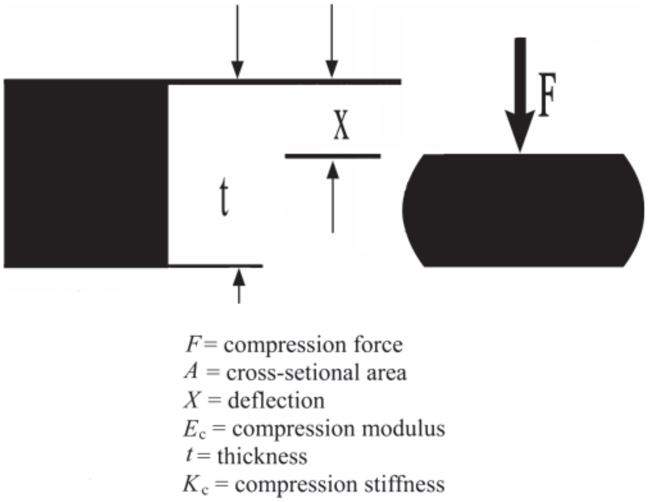

Fig. 1 Schematic view for compression test of rubber: $K_c = F/X = E_cA/t$

2.7 Thermal Aging of the Vulcanized Rubber

The vulcanized rubber specimen is heated at a temperature of 70 °C and is hung in an oven for 1, 3, 5, 7, and 30 days, respectively, to test its tensile strength, elongation at break, and also dynamic mechanical properties.

2.8 Dynamic Mechanical Analysis

A 2 mm thick vulcanized rubber specimen was cut to prepare a 2 × 10 × 5 mm specimen using dynamic mechanical analysis (DMA; TA-2980). The specimens were tested at a frequency of 1 Hz with an increment in the heating rate of 3 °C/min within a temperature range of −120 to 50 °C, to measure the storage modulus (E') and loss factor (tan δ) of the vulcanized rubber.

3. Results and Discussion

3.1 Mechanical Properties of NR/BR Blends

Different chemical structures of NR and BR show significant differences in the physical properties between vulcanized NR and BR rubbers. While being stretched on NR by applying stress across the arranged direction of its molecular chains, the molecular chains would develop an oriented (positive) arrangement under stress. This would then result in strain crystallization, thus improving its tensile strength (Ref 33). This shows that the mechanical properties of NR including tensile strength and elongation at break are clearly superior to those of BR. Figure 2 shows a tensile stress-strain curve of NR/BR blends. As can be seen, the tensile strain increases with increasing stress. The tensile stresses of NR and BR are 22.3 and 13.4 MPa, respectively, and their tensile strains are 1550% and 1350%, respectively. Both tensile stress and strain of NR are higher than those of BR. Moreover, the stress of NR increases drastically when the strain exceeds 1300%, meaning that the molecular chains develop strain crystallization when being stretched under stress. In NR/BR blends, with an NR content that is higher than that of BR, the domain of BR is distributed into NR. Therefore, the stress-strain curve tends to react in accordance with the tensile behavior of NR, so the stress-strain

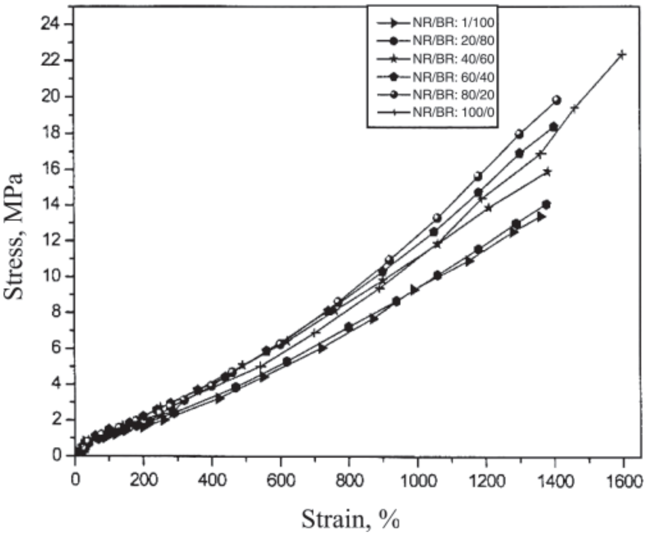

Fig. 2 Tensile stress-strain curve of NR/BR blends

Table 4 Tensile stress and tensile strain of NR/BR blends

	NR/BR ratio					
Properties	**0/100**	**20/80**	**40/60**	**60/40**	**80/20**	**100/0**
Tensile stress, MPa	13.4	14.0	15.9	18.2	19.8	22.3
Tensile strain, %	1350	1358	1360	1380	1410	1550

curve resembles that of NR. On the other hand, increasing the BR blend and decreasing NR content would make its tensile strength curve resemble that of BR. Because NR has the properties of strain crystallization, when its molecular chains are stretched under stress, the nonoriented molecular chains will then be arranged straight, which results in strain crystallization (Ref 26). Table 4 shows the tensile stress and tensile strain of NR/BR blends. Both the tensile stress and strain of NR are higher than those of BR. In addition, there is a tendency for both tensile stress and strain of NR/BR blends to be higher with increasing NR content. This means that both tensile stress and strain of BR would improve when blending NR into BR. Figure 3 shows a graph illustrating the relationship between NR content and tear strength of NR/BR blends. Factors affecting tear strength include the content of carbon black, chemical structure of the rubber itself, and fillers. The tearing behavior is influenced by the viscoelastic properties of the polymer. Moreover, there is a close relationship between the increase of tear energy and strain crystallization properties (Ref 34). As shown in the figure, the tear strength of NR and BR is 7.3 and 3.8 MPa, respectively. In addition, the tear strength of NR/BR blends tends to increase with increasing NR content. Moreover, the tear strength of NR/BR blends is similar to that of NR when NR content exceeds 80 phr.

3.2 Compression Stiffness and Compression Set of NR/BR Blends

The compressive deformation properties are directly affected by temperature. Thus, rubber material would have a softening effect and a degradation effect under high temperature. Meanwhile, a low-temperature condition will result in crystallization and hardening (Ref 35), thus causing a larger compressive deformation. Figure 4 shows the relationship between the composition and compression stiffness of NR/BR blends. The compression stiffness of NR is 5.5 kgf/mm, and that of BR is 10 kgf/mm. In other words, the compression stiffness of BR is about 2 times that of NR when subjected to the same deformation circumstances. Furthermore, the compression stiffness of NR/BR blends decreases with increasing NR content, meaning that, under compressive stress, NR not only has a higher load deformation than BR but is also more easily deformable. Figure 5 shows the relationship between NR content and compression set of NR/BR blends at 70 °C. As can be seen, the compression set of NR is 45%, and that of BR is 32%. The compression set of NR/BR blends increases with increasing NR content. A similar outcome was obtained with this compression set using a different blending ratio. Because the compression stiffness of NR is lower than that of BR, it also shows a lower relative elasticity with a softening effect caused by high temperature. The internal stress applied under compression resulted in the displacement of molecular chains due to the softening effect of rubber. Releasing the stress would generate a greater deformation due to its inability to recover the strain effectively.

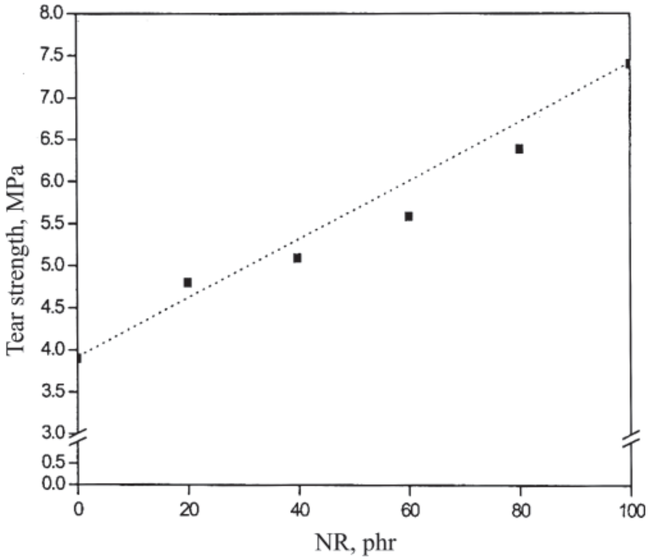

Fig. 3 Relationship between NR content and tear strength of NR/BR blends

3.3 Analysis of Air Permeability of NR/BR Blends

Table 5 shows the air permeability value of NR/BR blends. As can be seen, the measurement values, unit thickness permeability, and air permeability coefficient of BR are all higher than those of NR, implying that the air permeability of BR is higher than that of NR. In addition, the air permeability NR/BR blends tends to decrease with increasing NR content. This means that the air permeability of BR would improve when blending NR into BR.

3.4 Analysis of Physical Properties after Aging

Figure 6 and 7 show the tensile stress and strain curves of NR/BR blends at 70 °C, tested on different days. As can be

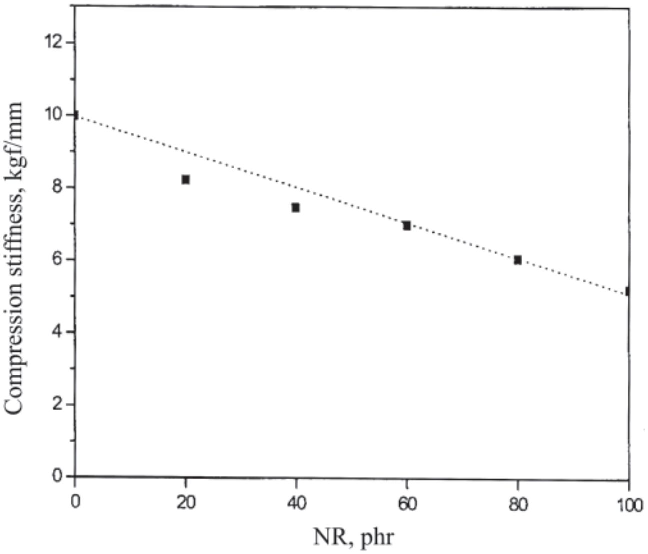

Fig. 4 Relationship between NR content and compression stiffness of NR/BR blends

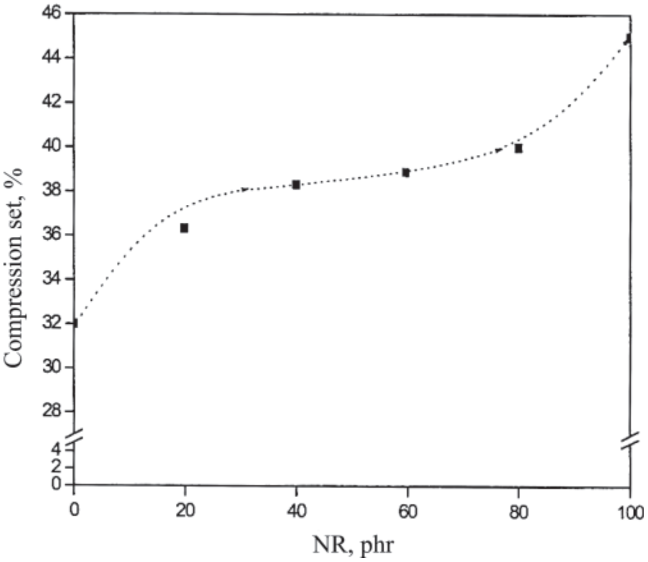

Fig. 5 Relationship between NR content and compression set of NR/BR blends at temperature of 70 °C

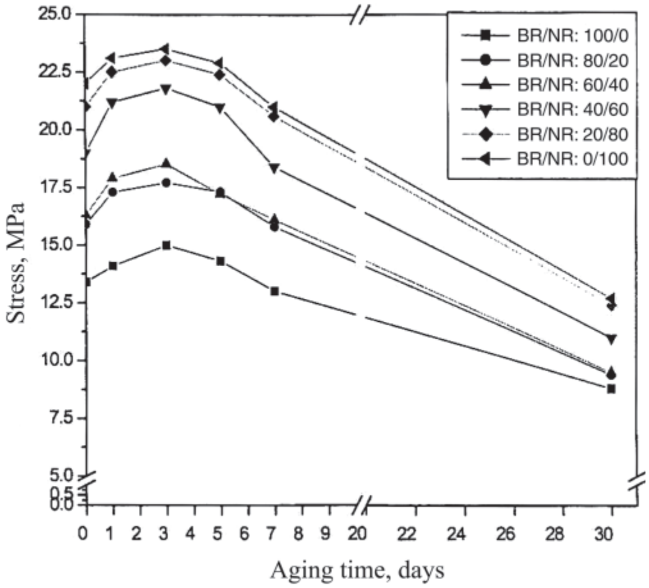

Fig. 6 Stress curve of NR/BR blends at a temperature of 70 °C when tested on different days

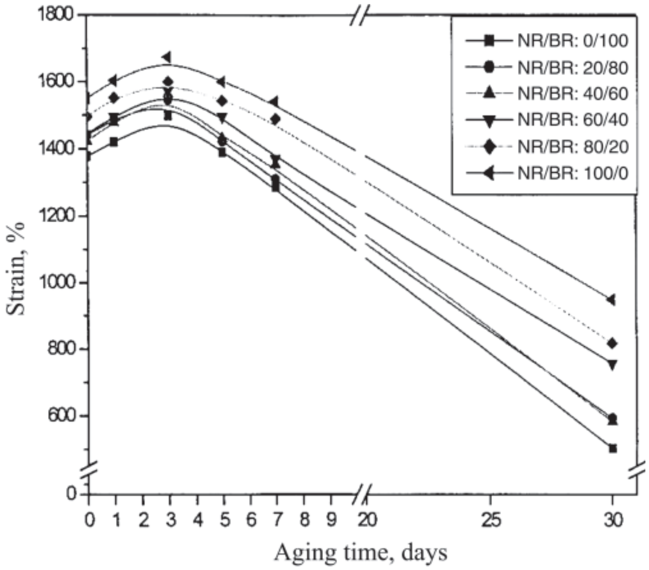

Fig. 7 Strain curve of NR/BR blends at a temperature of 70 °C when tested on different days

Table 5 Air permeability of NR/BR blends

	NR/BR ratio					
Testing items	**0/100**	**20/80**	**40/60**	**60/40**	**80/20**	**100/0**
1. Measured value, cm^3/m^2 24 h atm	414.46	383.77	370.44	358.03	343.70	271.60
2. Unit thickness permeability, cm^3 mm/m^2 24 h atm	825.11	785.33	717.01	598.04	590.74	450.85
3. Gas permeability coefficient, cm^3 cm/cm^2 s cm hg	1.25×10^{-9}	1.19×10^{-9}	1.09×10^{-9}	9.09×10^{-10}	8.98×10^{-10}	6.85×10^{-10}

seen, these stress-strain curves tend to rise initially and then decline. This means that a more complete cross-linking has occurred during the aging process of NR/BR blends at 70 °C. Thus, values of both tensile stress and tensile strain are high. With prolonged thermal aging, its aging resistance property become inferior due to the increased fractures in the cross-linked structure between the rubber molecular chains, resulting in lower tensile stress and strain. Tensile stress and strain curves of NR/BR blends aged for 30 days at 70 °C are shown in Fig. 8 and 9, respectively. As shown in the figures, the tensile stress and strain of aged NR are higher than those of BR. Moreover, values of both tensile stress and strain tend to increase with increasing NR content, similar to before aging. Figure 10 illustrates the stress loss of NR/BR blends after aging

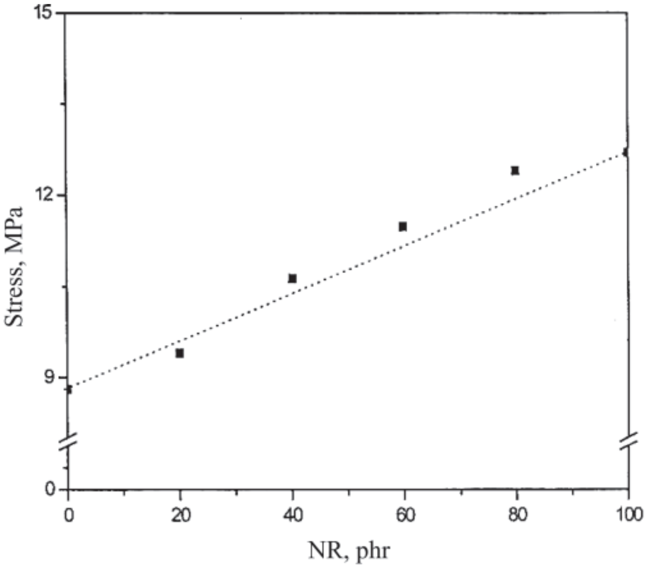

Fig. 8 Stress relationship graph of NR/BR blends aged for 30 days at 70 °C

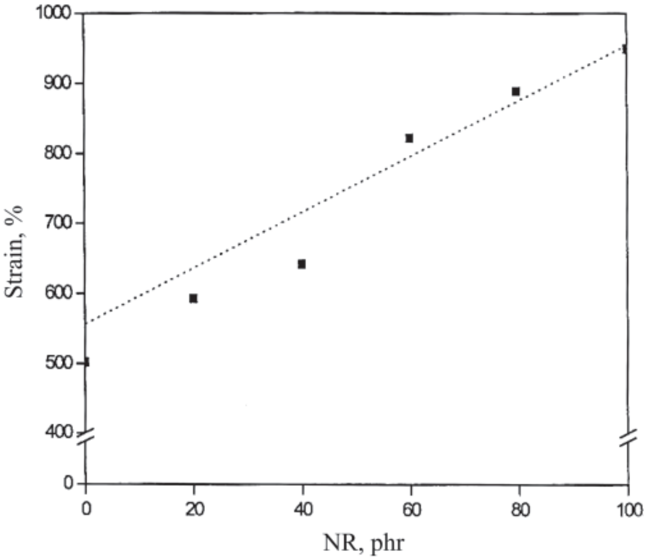

Fig. 9 Strain relationship graph of NR/BR blends aged for 30 days at 70 °C

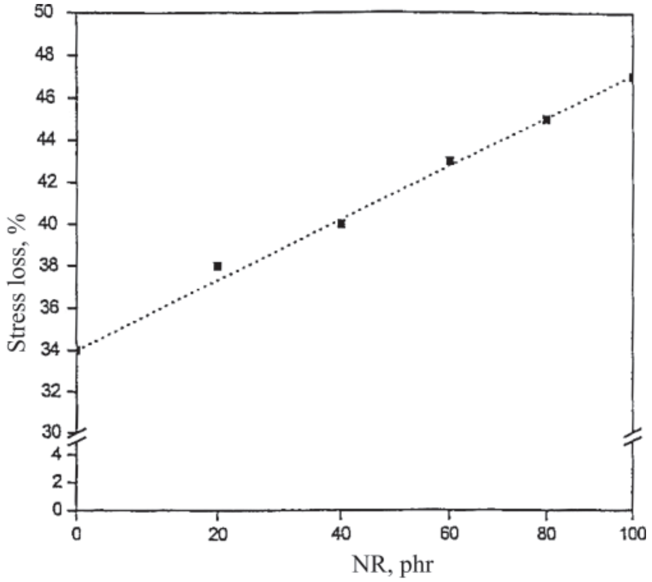

Fig. 10 Stress loss relationship graph of NR/BR blends after aging for 30 days

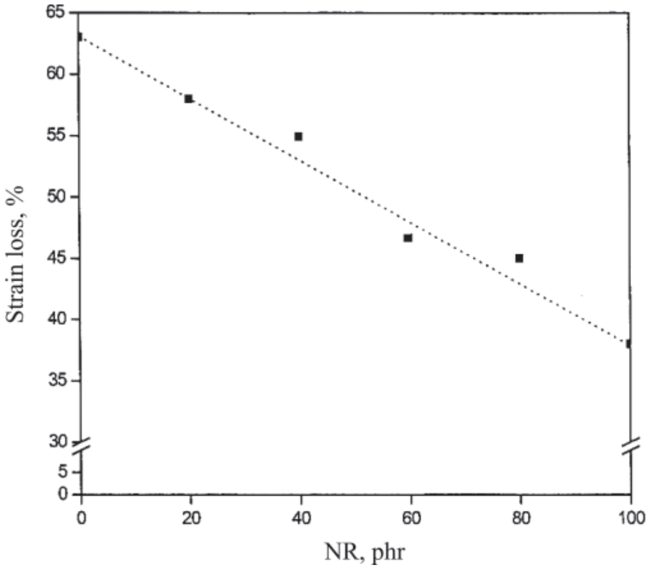

Fig. 11 Strain loss relationship graph of NR/BR blends after aging for 30 days

for 30 days. As can be seen, the stress loss of NR is higher than that of BR, meaning that BR loses less strength than NR during thermal aging. In addition, its stress loss increases with increasing NR content of NR/BR blends. BR also has better heat resistance than NR.

Figure 11 illustrates the strain loss of NR/BR blends after aging for 30 days. As can be seen, the strain loss of NR is less than that of BR and decreases with increasing NR content of NR/BR blends. This behavior is opposite to that of stress loss. When the stress loss of NR increases after prolonged thermal aging, only the elongation causes a lesser strain loss by a larger displacement of molecular chains resulted from a broken cross-linked structure. Figure 12 and 13 illustrate the changes in temperatures with the presence of a *T*g character peak after aging treatment of BR and NR, respectively at 70 °C. As can be seen, symbol Tα is the α-transition arising from segmental motion of molecular chains, this motion corresponds to the glass transition (Tg) of this rubber. This tendency is manifested three days before starting the aging process, and the cross-linking reaction of sulfur is further occured. The flexibility of the molecular chains is further limited, thus shifting the character peaks of $T\alpha_b$ (Tg character peak of BR) and $T\alpha_n$ (Tg character peak of NR) toward higher temperatures. Meanwhile, $T\alpha_b$ and $T\alpha_n$ tend to drop with prolonged thermal aging. In other words, prolonged thermal aging of NR/BR blends would achieve a gross motion, while there is a flexible motion in the molecular chains under low-temperature conditions due to the fractures in its cross-linked structure. Figure 14 and 15 show changes in the curves of BR character peak's tan δ_b (damping of BR) and NR character peak's tan δ_n (damping NR), with the aging process for NR/BR blends at 70 °C tested on different days. As can be seen, tan δ_b for BR and tan δ_n for NR tend to decrease initially with prolonged thermal aging, and then in-

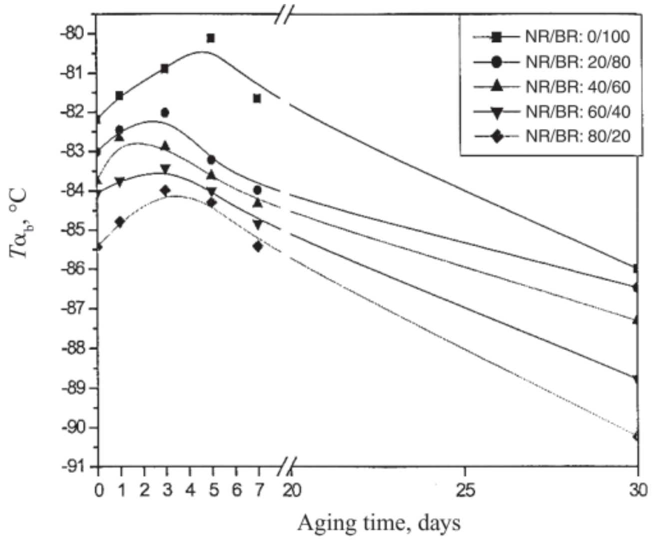

Fig. 12 Changes in temperature with the presence of a T_g character peak after aging treatment of BR at 70 °C

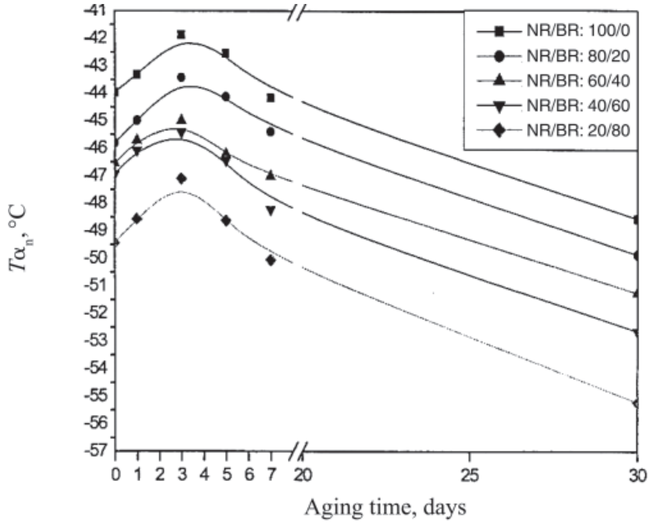

Fig. 13 Changes in temperature with the presence of T_g character peak after aging treatment of NR at 70 °C

crease, especially at higher BR content. Because the curing rates of NR are faster than those of BR, at about three days before the aging process begins, the thermal motion of the intermolecular chains is further limited due to cross-linking reactions. Thus, its damping characteristics decrease, and values of tan δ_b and tan δ_n also decrease. With prolonged thermal aging, the more fractures there are in the cross-linked structure in-between the rubber molecular chains, the easier the thermal motion in-between the molecular chains becomes. Therefore, tan δ_b for BR and tan δ_n for NR tend to increase with prolonged thermal aging.

4. Conclusions

In this work, the physical properties, air permeability, thermal aging, and dynamic mechanical properties of NR/BR blends were measured. From these results, the following conclusions were obtained:

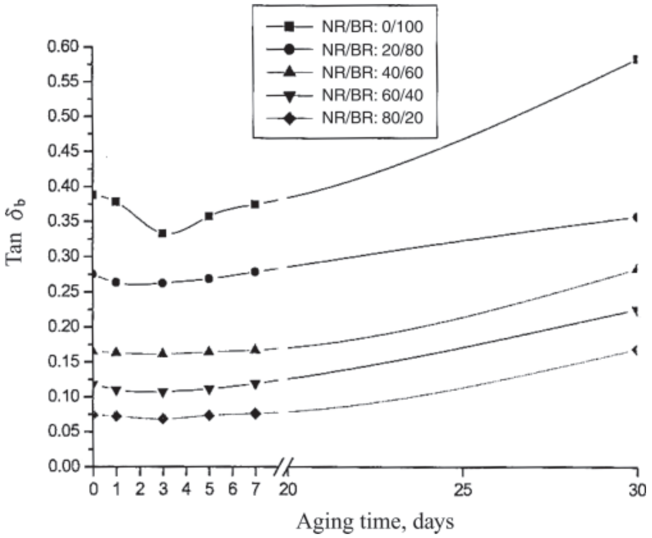

Fig. 14 Changes in tan δ_b, value of the BR character peak with an aging process for NR/BR blends at 70 °C tested on different days

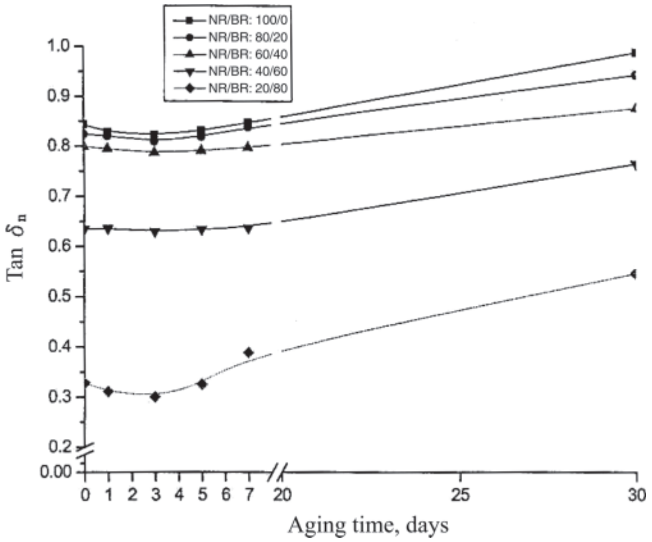

Fig. 15 Changes in tan δ_n, value of the NR character peak with an aging process for NR/BR blends at 70 °C tested on different days

- NR has higher tensile stress, tensile strain, and tear strength than BR. Moreover, both tensile stress and strain increase with increasing NR content of NR/BR blends.
- NR has higher a compression set than BR, and the compression set of NR/BR blends increases with increasing NR content. Meanwhile, the compression stiffness of NR is lower than that of BR, meaning that NR deforms more easily than BR.
- NR has lower air permeability than BR; air impermeability of NR is superior to that of BR. NR/BR blends become less gas permeable and achieve better air impermeability with increasing NR content.
- After prolonged thermal aging of NR/BR blends, both tensile stress and strain decrease. The stress loss of BR is also lower than that of NR, meaning that the aging resistance property of BR is superior to that of NR. With prolonged thermal aging of NR/BR blends, the more the fractures in

the cross-linked structure in-between the rubber molecular chains, the easier the thermal motion in-between the molecular chains becomes, thus shifting the T_g character peaks toward lower temperature range. Meanwhile, $\tan \delta_b$ and $\tan \delta_n$ tend to increase.

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