High Performance Elastomer Composites Containing Ultra High *cis* Polybutadiene with High Abrasion and Low Rolling Resistances

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ABSTRACT: A series of rubber composites with ultra high *cis* polybutadiene (UBR), NUC1–3, SBC1–4, SUS1–4, was prepared, and their vulcanized properties were measured and analyzed. The ultra high *cis* polybutadiene was prepared with the monomeric neodymium catalyst, Nd(neodecanoate)₃-(neodecanoic acid). NUC composites were composed of natural rubber, ultra high *cis* polybutadiene, and carbon black. In the composite of a low carbon black content (NUC1, 45 phr), a high abrasion-resistance and a significantly low rolling resistance (tan $\delta_{60^{\circ}C}$, 0.04) were obtained. According to the AFM study of NUC composites, abrasion resistance was closely related with surface morphology. In the SUS composites prepared with SSBR (solution styrenebutadiene copolymer), UBR, and silica, as the content of

INTRODUCTION

Reduction of rolling resistance, improvement of wet grip, and high abrasion-resistance are key subjects to develop high performance tire.¹ Recently, solution styrene–butadiene rubber (SSBR), starch, and silica technologies are introduced to achieve the state-of-the-art performance of tire.^{2–6} High abrasion resistance is one of the significant subjects due to economic and environment aspects, used to predict the service life of rubber products not only tire tread but also shoe soles, belts, and hose.^{7,8} Because of high oil price at present and in future, low rolling resistance is much required for fuel-saving.

Ultra high *cis* polybutadiene, having a very low T_g (ca. -110° C), is highly suitable to satisfy high abrasion resistance and low rolling resistance. Especially, the ultra high *cis* polybutadiene produced by monomeric neodymium catalyst performs excellent abrasion-resistance.⁹ However, UBR composite study was not much carried out in the blends with natural rubber (NR), styrene–butadiene rubber (SBR) or SSBR for the purpose of reduction of heat build-up,

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ultra high *cis* polybutadiene increased, Δ cure torque (M_{H^-} M_L) increased with fast cure kinetics. SUS4 showed high elongation and tensile strength with excellent abrasion resistance. Rolling resistance was improved as the content of ultra high *cis* polybutadiene increased. The SBC composites were prepared with SBR (emulsion styrene–butadiene copolymer), ultra high *cis* polybutadiene (or high *cis* polybutadiene), and carbon black. It is remarked that abrasion resistance and Δ tan δ (tan $\delta_{60^{\circ}C}$ – tan $\delta_{0^{\circ}C}$) are increased with ultra high *cis* polybutadiene. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 105: 477–485, 2007

Key words: ultra high *cis* polybutadiene; abrasion-resistance; rolling-resistance

decrease of rolling resistance, and increase of abrasion resistance in the balance of wet-skid resistance.

Our aim in this study is to design high performance composites and examine the surface structures to understand abrasion- and rolling-resistance properties.

EXPERIMENTAL

Materials

Ultra high *cis* polybutadiene (UBR: Nd-BR 40, neodymium-catalyzed polybutadiene, cis content 98%) and high *cis* polybutadiene (BR: KBR-01, nickelcatalyzed polybutadiene, cis content 96%), SBR (SBR1712: Styrene 23.5%, oil 37.5 phr, MV = 49), and SSBR (SSBR6360S: Styrene 30%, oil 37.5 phr, MV = 53) were obtained from Korea Kumho Petrochemical (Seoul, Korea).

Formulation

The compound recipes varying by NR, SBR, SSBR, BR, and UBR are summarized in Tables I–III.

Compounding

Rubbers, carbon black, oil, zinc oxide, and stearic acid were placed in a 3-L-kneader. In the first

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TABLE I NUC Compounding Component

Component (phr)	NUC1	NUC2	NUC3
Natural rubber (STR-20)	70	70	70
UBR (Nd-BR)	30	30	30
Carbon black (N-220)	45	50	64
Zinc oxide	4	4	4
Stearic acid	1.5	1.5	1.5
Sulfur	2.2	2.2	2.2
Accelerator NS	0.9	0.9	0.9

mixing step, the rubber mixture was kneaded for 5.5 min at 120°C and 45 rpm. In the second mixing step, the compound, sulfur, and activator were mixed for 3 min at 70°C and 45 rpm.

Characterization

The microstructure of polybutadiene was measured in CS_2 solution by infra-red spectroscopy (Bio-Rad, FTS 60-A; Hercules, CA) according to the literature.¹⁰ Gel permeation chromatography data were obtained using a Viscotec system (TDA 300; Houston, TX) employing connected TOSOH columns (G6000HHR, G5000HHR, G4000HHR, G3000HHR) with a refractive index detector. Tetrahydrofuran was used as solvent at the flow rate of 1.0 mL/min.

Bound rubber measurement

A piece of compounded rubber (1 g) was cut into pieces of about 2 mm size, placed in a flask, and toluene (200 mL) was added. The flask was shaken for 48 h at room temperature. The insoluble rubber was filtered, dried, and weighed.

Compound property

Mooney viscosities of raw polymers and their compounds were measured at 100°C with a Mooney MV 2000 of Alpha Technologies (Akron, OH). Physical

TABLE II SUS Compounding Component

Component (phr)	SUS1	SUS2	SUS3	SUS4
SSBR	117	110	103	96
UBR (Nd-BR)	15	20	25	30
Silica	50	50	50	50
Si69	4	4	4	4
Processing oil	6.3	8.1	10	11.9
Zinc oxide	4	4	4	4
Stearic acid	2	2	2	2
Sulfur	1.5	1.5	1.5	1.5
Accelerator				
CZ	1.5	1.5	1.5	1.5
DPG	1.0	1.0	1.0	1.0

TABLE III SBC Compounding Component

Component (phr)	SBC1	SBC2	SBC3	SBC4
SBR	103	103	103	103
BR (Ni-BR)	25	25		
UBR (Nd-BR)			25	25
Carbon black (N-375)	80	70	80	70
Processing oil	10	10	10	10
Zinc oxide	3	3	3	3
Stearic acid	2	2	2	2
Sulfur	1.8	1.8	1.8	1.8
Accelerator				
CZ	1.3	1.3	1.3	1.3
DPG	0.3	0.3	0.3	0.3

properties of the compounds were measured with a universal testing machine (Instron 6021; Norwood, MA). Dynamic mechanical analysis was employed to characterize the compounds using a Rheometric ScientificTM Model DMTA V (Piscataway, NJ). Abrasion tests were carried out according to DIN 53516 with a DIN abrasion tester of Bareiss (Oberdischingen, Germany). The test sample was of a good cylindrical shape with a diameter of 16 \pm 0.2 mm. A rubber process analyzer (RPA 2000, Alpha Technology) was used for studying the macrostructure of polymer.

Surface morphology

The surface morphology of the composites of NUC and SBC was investigated by tapping mode of a scanning probe microscope (Shimadzu SPM-9500J3; Tokyo, Japan) with a silicon nitride tip. Scanning electron microscopy (SEM) images were collected using a JEOL JSM-5400 (Tokyo, Japan). Samples were sputter-coated with gold prior to examination.

RESULTS AND DISCUSSION

NUC composites

To obtain ultimate abrasion-resistance, NUC composites were prepared with natural rubber (NR), ultra high cis polybutadiene, and carbon black. In Tables I and IV, the amount of carbon black was varied from 45 to 64 phr (per hundred rubber weight) and the blend ratio of NR and UBR was 70 to 30 (phr/phr), respectively. Natural rubber, which possesses high physical properties such as tensile strength, resistance to tear, cut, and chip, is the base polymer for NUC composites.^{2,11–15} However, NR is wearable. To increase abrasion-resistance, ultra high cis polybutadiene, prepared by monomeric neodymium catalyst, was employed with high structural carbon black N220.16 Composite density, compound viscosity, and cure torque $[M_L, M_H, \Delta torque (M_H-M_L)]$ increased, and cure kinetics (T_{90} , 14.3 min) was fast as carbon black increased. As for NUC1 and NUC2 composites

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	NUC1	NUC2	NUC3
Carbon black amount (phr)	45.0	50.0	64.0
Density	1.111	1.139	1.169
Comp. Mooney viscosity, (ML ₁₊₄ , 100° C)	87.7	96.9	>110
Cure properties (145°C)			
M_L (dNm)	5.88	6.65	10.10
M_H (dNm)	37.1	39.1	50.8
T_{10} (145°C) (min)	8.3	8.1	6.7
T ₅₀ (145°C) (min)	11.0	10.8	9.3
T ₉₀ (145°C) (min)	16.7	16.0	14.3
Tensile properties (ASTM D412)			
Hardness (Shore A)	66	68	74
100%-Modulus (kgf/cm ²)	34.7	39.6	58.7
200%-Modulus (kgf/cm ²)	90.8	105.6	149.4
300%-Modulus (kgf/cm ²)	165.4	186.4	_
Tensile Strength (kgf/cm ²)	240.5	236.8	233.2
Elongation at break (%)	394.6	362.8	296.6
Tear strength (kgf/mm)	7.20	7.53	8.08
Abrasion (DIN) weight loss (g)	0.131	0.131	0.139
DMTA (1.66 Hz/0.02%)			
T_{g} (°C)	-54.8	-54.5	-53.8
tan δ (0°C)	0.127	0.120	0.162
$\tan \delta (60^{\circ}C)$	0.042	0.048	0.093

 TABLE IV

 NUC Compound Results with Ultra High cis Polybutadiene

with low carbon black contents (45, 50 phr), proper compound viscosity, high tensile strength, and elongation-at-break values were obtained. The composite with high carbon black content (NUC3, 64 phr) yielded high compound viscosity, modulus, tear strength (8.08 kgf/mm), and antiwet-skid (tan $\delta_{0^{\circ}C}$, 0.162) values, but low tensile and dynamic properties such as relatively low abrasion resistance (0.139 g) and high rolling resistance (tan $\delta_{60^{\circ}C}$, 0.093) were obtained. Although tear strength is closely related with carbon black content, poor processibility can be expected by high compound viscosity and hardness. Tensile strength (233–240 kgf/cm²) and 300%-modulus (165–190 kgf/cm²) of NUC composites were much higher than those of SUS and SBC composites in Tables V and VI. These results are attributed to the strain-induced crystallization of natural rubber, high surface area of carbon black, and high content of UBR. The high abrasion resistance is explained by high hardness, tensile strength, and elongation properties: *A* (abrasion) ~ $\mu/H\sigma\epsilon$ (μ : traction force, *H*: hardness, σ : tensile strength, ϵ : elongation-atbreak).¹⁷ In these aspects, the abrasion-resistance order is expected as NUC1 > NUC2 > NUC3. As

 TABLE V

 SUS Compound Results with Ultra High cis Polybutadiene

	SUS1	SUS2	SUS3	SUS4
Ratio (SSBR/UBR)	85/15	80/20	75/25	70/30
Comp. Mooney viscosity (ML ₁₊₄ , 100° C)	89.9	89.3	88.9	91.1
Cure properties (145°C)				
M_L (dNm)	8.39	8.45	8.13	7.99
M_H (dNm)	26.47	26.79	27.18	27.55
T_{10} (min)	15.0	15.4	15.4	14.5
T_{50} (min)	22.9	23.1	22.7	21.7
T_{90} (min)	33.0	32.7	31.9	30.6
Tensile property (ASTM D412)				
Hardness (Shore A)	64	64	64	64
100%-Modulus (kgf/cm ²)	21.7	22.0	20.7	20.9
200%-Modulus (kgf/cm ²)	41.5	42.6	39.9	40.6
300%-Modulus (kgf/cm ²)	63.1	65.8	61.0	62.6
Tensile strength (kgf/cm^2)	125.9	125.1	116.1	131.3
Elongation break (%)	510.1	491.3	491.2	520.9
Abrasion (DIN) weight loss (g)	0.229	0.237	0.225	0.186
DMTA (1.66 Hz/0.02%)				
T_{g} (°C)	-12.0	-13.0	-14.5	-17.2
tan δ (0°C)	0.584	0.468	0.401	0.343
tan δ (50°C)	0.105	0.115	0.092	0.097

SBC Compound Results with Oltra High <i>cis</i> Polybutadiene						
	SBC1	SBC2	SBC3	SBC4		
Ratio (SBR/BR/CB)	75/25/80	75/25/70	75/25/80	75/25/70		
BR cis content (%)	95	95	98	98		
Bound rubber content (%)	53	44	46	43		
Comp. Mooney viscosity,	68.2	61.9	72.6	61.2		
(ML ₁₊₄ , 100°C)						
Cure properties (145°C)						
M_L (dNm)	4.00	3.67	4.23	3.52		
M_H (dNm)	25.23	24.50	25.77	23.24		
T_{10} (min)	9.0	8.7	6.6	8.0		
T_{50} (min)	11.0	11.7	9.3	11.0		
T_{90} (min)	17.4	18.5	15.3	17.7		
Tensile property (ASTM D412)						
Hardness (Shore A)	62	61	62	57		
100%-Modulus (kgf/cm ²)	24.0	22.3	27.7	21.8		
200%-Modulus (kgf/cm ²)	60.7	56.4	71.4	54.3		
300%-Modulus (kgf/cm ²)	108.7	103.2	128.2	100.5		
Tensile strength (kgf/cm ²)	180.5	186.8	199.9	199.0		
Elongation at break (%)	450	476	433	501		
Tear strength (kgf/mm)	6.49	6.43	6.53	6.45		
Abrasion (DIN) weight loss (g)	0.173	0.168	0.164	0.150		
DMTA (1.66 Hz/0.02%)						
tan δ (0°C)	0.124	0.134	0.128	0.148		
tan δ (60°C)	0.113	0.113	0.111	0.117		

 TABLE VI

 SBC Compound Results with Ultra High cis Polybutadiene

the result of DIN abrasion test, the abrasion-resistance order was obtained as NUC2 > NUC1 > NUC3. The abrasion-resistance (0.131–0.139 g) was much higher than those of SUS (0.186–0.237 g) and SBC (0.150– 0.173 g) composites, which are attributable to the high tensile and modulus properties of NUC. The ratio of loss over storage modulus G''/G' (tan δ), indicates the dynamic property and network structure of elastic materials for their hysteretic energyloss processes, such as rebound, heat generation, and wet-skid.^{18–20} The dynamic property at the temperature region of 60°C represents rolling resistance and rebound, and at 0°C represents the wet traction of tire. NUC1 and NUC2 showed low tan δ values (0.042, 0.048), which indicate significantly low rolling resistance with solid filler networks. NUC3 yielded a high tan δ , indicating high antiwet-skid property and rolling resistance. Topological atomic force microscopy (AFM) images of NUC1-3 obtained by cyrocutting with a microtomb are shown in Figure 1, which provide topological microphases such as filler dispersion on rubber matrix and rubber surface morphology.²¹ As for the AFM image of NUC1, a homogenous and smooth surface was observed, and carbon black was well dispersed in rubber matrix. With regard to the AFM image of NUC3, discrete domains were clearly observed. Higher carbon black content resulted in higher segregated domains.



Figure 1 Topological AFM data of NUC composites.

As for the NUC2 image, continuous and discrete domains exist together. It is remarked that surface morphology is closely related with abrasion and rolling resistance. The continuous surface (NUC1) results in high abrasion-resistance and low rollingresistance, while isolated domains (NUC3) yield high wear and rolling-resistance. NUC1 and NUC2 composites are suitable for high abrasion-resistance and low rolling-resistance applications.

SUS composites

Silica compounding is employed to improve the trade-off relationship between wet traction and rolling in the tire application.²² In Tables II and V, SUS1-4 were the silica compounds of solution-SBR and ultra high cis polybutadiene. The solution SBR is an oil-extended grade, and the microstructure and GPC data are as follows: styrene 30%, vinyl 48%, M_{ν} 750,000, and MWD 1.75. The oil content was varied according to SSBR amount used. The compound viscosity of SUS series was in the range of 90 MU, and the Δ cure torque (M_H - M_L) of SUS4 was a little high (SUS1 18.08, SUS4 19.56). The cure kinetics $[T_{10}]$ (min): 14.5-15.4] was much slower than those of NUC [T₁₀ (min): 6.7–8.3] and SBC [T₁₀ (min): 6.6–9.0] series, attributed to silica compounding.²² Silica undesirably retards the cure rate. It is notable that with an increase of ultra high cis polybutadiene, the cure time was shortened [T_{90} (min): SUS1, 33.0; SUS4, 30.6]. Even at the same hardness values as SUS1-3, high tensile, elongation, and dynamic properties were observed in SUS4, which are attributed to ultra high cis and linear structure of polybutadiene. Antiwet-skid property (tan $\delta_{0^{\circ}C}$, 0.584–0.343), one of the most important factors for automobile safety, was significantly larger than those of carbonfilled NUC and SBC (0.124–0.162). The high antiwetskid property resulted from the silica compounding with SSBR consisting of high vinyl and styrene contents.

SBC composites

Ultra high *cis* polybutadiene blended with SBR was employed to balance both abrasion-resistance and antiwet-skid properties. In Table VI, as for SBC1 and SBC2, high *cis* polybutadiene (Ni-BR, *cis* content 95%) was used, and for SBC3 and SBC4, ultra high *cis* polybutadiene (Nd-BR, *cis* content 98%) was employed, respectively. Neodymium-polybutadiene has ultra high *cis*, linear, and narrow MWD, showing high elastic properties in vulcanized condition, while nickel-polybutadiene with 95% cis content, branched structure, and a broad MWD shows relatively high viscositic properties.^{9,23} Figure 2 shows the tan δ values of uncured Ni- and Nd-polybuta-



Figure 2 Comparison of Ni-polybutadiene (BR) and Nd-polybutadiene (UBR) with RPA frequency sweep (100°C, 7% strain).

diene from RPA tests (at 100°C, 7% strain) to account for the viscoelastic character. A high degree of branching and a broad MWD of Ni-polybutadiene show a low tan δ value due to high entanglement, while Nd-polybutadiene yields a high tan δ . The bound rubber content was increased in the order SBC1 > SBC3 > SBC2 > SBC4. Relatively high vinyl, broad MWD and branched Ni-polybutadiene more strongly interacts with carbon black than Nd-polybutadiene does.²⁴ Cure time was also related with carbon content and polybutadiene structure. High carbon black content and ultra high cis polybutadiene showed fast cure kinetics (SBC3, $T_{10}(min)$ 6.6, $T_{90}(\text{min})$ 15.3). As the contents of carbon black and ultra high cis polybutadiene increased, compound viscosity became increased, and as expected, high tear strength was obtained. High compound viscosity caused by the molecular structure of ultra high cis polybutadiene was decreased by reducing the amount of carbon black in SBC4, and high tensile, elongation, and dynamic properties were also obtained. High elongation and tensile properties of SBC4 gave rise to high abrasion-resistance. This result is attributed to the solid crosslinking network made by the linearity and narrow MWD of ultra high *cis* polybutadiene. It is also noted that high tan δ (at 0°C, 0.148) and $\Delta \tan \delta$ (tan $\delta_{60^{\circ}C}$ – tan $\delta_{0^{\circ}C}$) values were obtained. Ultra high cis polybutadiene blended with SBR provides high abrasion-resistance and well-balanced antiwet-skid properties. Topological atomic force microscopy (AFM) images of SBC1-4 obtained by cyrocutting are shown in Figure 3. As for the AFM image of SBC1, a coarse and rough surface was observed, and carbon black was poorly dispersed in the rubber matrix. With regard to the AFM image of SBC2, carbon black was better dispersed than that of SBC1 due to a low amount of carbon black. As for SBC3, coarse and segregated domains were found. The well-dispersed surface morphology was obtained in the AFM image of SBC4, where carbon black was well embedded in the rubber matrix. The SEM micrographs of the worn



Figure 3 Topological AFM data of SBC composites.

surface after the DIN abrasion tests of SBC1-4 are shown in Figure 4, which corresponded well to the AFM images of SBC1-4. Coarse and small fragments were observed in the SEM images of SBC1-3, and the structure of the worn surface was a mixture of flake and rod-types. During abrasion, the surface was flaked or tore away as small bits or pieces. A remarkable difference compared with SBC1-3 was found in the image of SBC4. The worn surface of SBC4 was smooth and little wear debris was formed. From the observation of AFM and SEM, the surface of SBC4 before and after abrasion test was smoother than those of SBC1-3, which is one of the important factors to result in high abrasion-resistance.

SBC composites were lower than those of NUC and

SUS composites (88-100 MU). NUC composites show the highest modulus, tensile strength, and abrasion, but low elongation and tan δ values, which are suitable for high abrasion- and low rolling-resistance. SUS composites provide the highest elongation and tan δ values, but low properties in modulus, tensile strength, and abrasion. SBC composites show moderate performances in tensile and dynamic

Comparison of NUC, SUS, and SBC composites

Figure 5 makes the comparison of the average physical properties of the series of NUC, SUS, and SBC

composites. The compound viscosities (61-73 MU) of



Figure 4 SEM micrographs of the worn surface of SBC1–4.

properties. In the comparison of SBC composites, SBC-U, prepared with ultra high *cis* polybutadiene, shows better performances in the physical properties of modulus, tensile strength, and abrasion resistance than SBC-C, prepared with high *cis* polybutadiene. It is also noted that even wet-skid resistance is higher and T_{10} (min) is shorter than those of SBC-C.

CONCLUSIONS

Ultra high *cis* polybutadiene, prepared by the monomeric neodymium catalyst, Nd(neodecanoate)₃·(neodecanoic acid), brings some important impacts on high performance composites. Below is the summary of the results:

1. Surface morphology is strongly related with abrasion-resistance. A continuous and homogenous surface shows better high abrasion-resistance than coarse or segregated one (NUC1 and SBC4). The highly abrasive surface (SBC1) is of flake and rod-type. The worn surface of high abrasion-resistance composite (SBC4) was smooth and little wear debris was formed. Abrasion resistance of NUC composites is higher than those of SBC and SUS composites.

- 2. The difference of tan δ , $\Delta tan \delta$ (tan $\delta_{60^{\circ}C}$ tan $\delta_{0^{\circ}C}$), is maximized in the recipe of ultra high *cis* polybutadiene with a low carbon content. A significantly low rolling resistance value (tan $\delta_{60^{\circ}C}$, 0.04) was obtained in continuous and homogeneous surface (NUC1).
- 3. High carbon composites result in coarse or segregated surface structure, which yields low abrasion resistance and high rolling resistance.
- 4. Slow cure kinetics due to silica compounding is improved by increase of ultra high *cis* polybuta-diene.
- While silica composites (SUS) present high antiskid property, carbon composites (NUC, SBC) bring better abrasion resistance than silica composites do.



Figure 5 Comparison of the physical properties of NUC, SUS, and SBC composites.

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