

Study on the Structure-Mechanical Properties Relationship and Antistatic Characteristics of SSBR Composites Filled with SiO₂/CB

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ABSTRACT: The composites of solution polymerized styrene-butadiene rubber (SSBR) filled with SiO₂/carbon black (CB) were prepared by reaction blending. The mechanical and antistatic properties were studied. The morphology and dispersion of the two kinds of nano-fillers in the composites were observed. The results showed that CB presented 30 nm spherical particles and formed filler networks in the SSBR/CB composite. Payne effect of the SSBR/CB composite was large and the antistatic property was excellent; SiO₂ powder presented 20–40 nm irregular particles and also formed filler networks in the SSBR/SiO₂ composite. The internal friction of the SSBR/SiO₂ composite was low, however, the static accumulation was obviously large; SSBR/

SiO₂/CB composites exhibited good filler dispersion, high mechanical properties and low internal friction. The rolling resistance, wet-skid resistance and wear resistance are well balanced. Among these, the composite filled with SiO₂/CB in 20/50 achieved the best overall performances. The surface and volume resistivity of the composites was increased with increasing amount of SiO₂. For the researched formulation, the ratio of SiO₂/CB in 35/35 was the percolation threshold for the antistatic property of the composites. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 118: 338–345, 2010

Key words: dispersions; elastomers; glass transition; morphology; silicas

INTRODUCTION

In response to ever more demanding requirements from the environmental protection and social sustainable development, the automobile performances are increasing year after year. Thus the requirements for green tires with high wear resistance, low rolling resistance, and excellent wet-skid resistance were stricter and stricter.¹ The three performances are hard to be improved simultaneously, (i.e., one or two properties are improved whereas another two or one property will be decreased), which are often called “magic triangle” in the tire industry.² Accordingly, it is a hard problem for the researchers all over the world to develop an ideal tread compound to balance the “magic-triangle” properties and meet the “green tire” standards, (i.e., low loss, high security, and long service life).³ In recent years, many kinds of novel rubbers used in the tire tread have constantly emerged, together with their nanometer fillers,^{4,5} and a reaction blending technology has also been adopted.

Carbon black (CB) is a kind of nano-filler made from the incomplete combustion of the polycyclic aromatic hydrocarbon. The fundamental plane of CB particles consists of the unsaturated hexatomic rings and is arranged concentrically. As the electron on the hexatomic-ring plane can flow, CB has the anti-static property. High structure and small particle size CB, such as acetylene carbon black and intermediate super abrasion furnace black (ISAF) has good reinforcing performances and electrical conductivity.^{6–9} Due to using CB as the tire tread filler, electrostatic charge is easy to be exported. Researching the antistatic problem of tires has not been put on the agenda for many years.

In recent years, nano-SiO₂ powder has been greatly used in the “green tire” tread, which plays an important role in balancing the “magic-triangle” properties,^{10–13} especially in decreasing rolling resistance.^{14,15} However, nano-SiO₂ powder is made up of the Si—O tetrahedron, on which the electron could hardly flow. Thus the insulating property is presented. When CB in the tire tread is partly replaced by nano-SiO₂ powder, the tire and even the car will be easy to accumulate more static. Thus discharges will occur as soon as the tires or the cars contact the conductor, which would lead to the fire disaster and explosion of the oil tank in the gas station etc.¹⁶ However, solving the static problems caused by the tire tread filled with nano-SiO₂

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powder is another critical issue for the tire's safety today, which has rarely been reported.

Therefore, the purpose of this work was the study on the structure-mechanical properties relationship and antistatic characteristics of solution polymerized styrene-butadiene rubber (SSBR) composites filled with SiO₂/CB, which were prepared through thermal reaction blending technique. The mechanical properties, compression heat build-up and rolling power loss of the tire tread composites were investigated. The surface and volume resistivity was introduced to characterize the antistatic property of the rubber compound, and the relationship between the morphology of the fillers and the antistatic property of the composites was also discussed. The antistatic percolation threshold of the SSBR composites filled with SiO₂/CB was presented. It is expected that these experimental results could provide some theoretical and practical basis for the design and manufacture of high performance tires with both safe and energy-saving properties.

EXPERIMENTAL

Materials

SSBR (YL950), [$M_n = 1.23 \times 10^5$ g/mol, $M_w = 2.58 \times 10^5$ g/mol, molecular weight distribution (MWD) = 2.1, styrene content (%) = 27.5, vinyl content (%) = 25.4] was produced by Academe attached to Yanshan Petrochemical (Beijing, China); CB (N234) was purchased from Tianjin Haitun Carbon Black (Tianjin, China); The precipitated silica (Tixosil 383) was produced by Rhodia Silica Qingdao (Qingdao, China); Bis [γ -(triethoxysilyl) propyl] disulfide (Si-75) was purchased from Jingzhou Jiangnan Fine Chemical (Jingzhou, China); Other materials were commercial products.

Formulation and the sample coding

Formulation

The formulations of SSBR filled with different ratios of SiO₂/CB are listed in Table I.

The sample coding

The samples are identified by SSBR filled with SiO₂/CB in the ratio 0/70, 20/50, 35/35, 50/20, and 70/0, respectively.

Specimen Preparation

Preparation of the rubber compounds

SSBR was put onto an open two-roll mill (Shanghai Rubber Machinery Works No. 1, Shanghai, China), and then nano-SiO₂ powder and Si-75 were added into the rubber compounds to be mixed. The compounds were placed at room temperature for 1 day. And then reaction blending was done by a Haake PolyLab torque

TABLE I
Recipes of the SSBR Filled with Different Ratios of SiO₂/CB

Ingredients	Loading (phr) ^a
SSBR	100.0
SiO ₂ /CB	Variable ^b
Si-75	Variable ^c
Zinc oxide ^d	4.0
Stearic acid ^e	2.0
Antioxidant ^f	1.0
Accelerator Cz ^g	1.5
Accelerator D ^h	Variable ⁱ
High aromatic oil ^j	20.0
Sulfur ^k	1.8

^a Parts-per-hundred rubber.

^b Keeping the overall content of fillers 70.0 phr, which is widely used as reinforcing agents in the formulation of the green tire tread currently. The ratios of SiO₂/CB are 0/70, 20/50, 35/35, 50/20, and 70/0, respectively.

^c Si-75 is used to improve the affinity between SiO₂ and SSBR, and the mass fraction of Si-75 is 7% of the mass of the nano-SiO₂ powder.

^d Vulcanization activator.

^e Vulcanization activator and plasticizer.

^f *N*-Isopropyl-*N'*-phenyl-*p*-phenylene Diamine.

^g *N*-cyclohexyl-2-benzothiazyl Sulfenamide, vulcanization accelerator.

^h Diphenyl Guanidine, vulcanization accelerator.

ⁱ The mass fraction of Accelerator D is 2.5% of the mass of the nano-SiO₂ powder.

^j The plasticizer, to improve the processability of the rubber.

^k Curing agent.

rheometer equipped with a Rheomix 610p mixing chamber and roller rotors (Thermo Electron Corporation, Waltham, MA) at a temperature of 150°C for 4 min. The temperature was controlled by changing the speed of rotor. The mixing curves are shown in Figure 1. After the compounds were cooled down, CB and other addition agents were added successively into the rubber compounds by the two-roll mill. Finally the rubber compounds were sheeted for future use.

Preparation of vulcanizates

The vulcanizing properties were determined at 150°C by a P3555B₂ Disc Vulkameter (Beijing Huanfeng Chemical Machinery Trial Plant, Beijing, China). And then the vulcanizates were prepared by a XLB-D350 × 350-type Automatic Operation Vulcanizing Press (Huzhou Dongfang Machinery, Zhejiang, China). The preparation condition of vulcanizates was 150°C × t_{90} (optimum cure time) and the hydraulic pressure was 15 MPa.

Characterizations

Microstructure and morphology

Microstructure and morphology was observed by H-800-1-type Transmission Electron Microscopy (TEM),

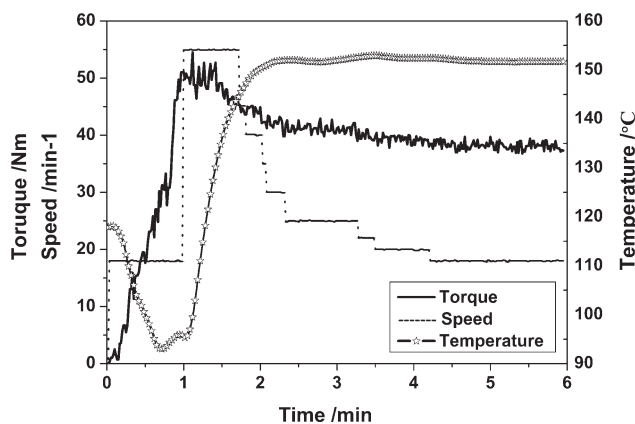


Figure 1 The mixing curves of Haake Torque Rheometer (the amount of SiO_2/CB is 35/35).

produced by Japan Hitachi, Tokyo, Japan. The acceleration voltage was 200 kV. The thin sections of the samples were cut by microtome under -100°C and collected on the cooper grids.

Mechanical properties

Mechanical properties were tested at $25^\circ\text{C} \pm 2^\circ\text{C}$ according to ASTM D638 by CMT4104 electrical tensile tester produced by Shenzhen SANS Test Machine, Shenzhen, China, at a tensile rate of 500 mm/min. The test area of the dumbbell-shaped tensile specimen is $25 \times 6 \times 2$ mm and the nicked angle tear specimen is $100 \times 19 \times 2$ mm. Shore A hardness of the vulcanizates was measured with a rubber hardness apparatus produced by the fourth Chemical Industry Machine Factory, Shanghai, China.

Wearability

Wearability was determined according to GB/T 1689-82 by MZ-4061 Akron abrasion machine, produced by Mingzhu Testing Machinery, Jiangdu, China. The rotational speed of the rubber wheel spindle was 76 rpm, the acting force on the rubber wheel was 26.7 N and the tilt angle between the rubber roll spindle and the grinding wheel spindle was 15° . The test was conducted at room temperature.

Dynamic compression fatigue heat build-up

Dynamic compression fatigue heat build-up was determined according to GB/T 1687-1993 by YS-25 Compression Fatigue Tester, produced by Shanghai rubber machinery works, Shanghai, China. Testing conditions: the preheating time was 20 min, the compression time was 25 min, the frequency was 1800 min^{-1} , the stroke was 4.45 mm and the testing load was 1 MPa.

Dynamic mechanical property (strain sweep)

Dynamic mechanical property (strain sweep) was determined by RPA2000 rubber process analyzer, produced by Alpha Technologies, OH. The testing temperature was 60°C , the strain was varied from 0.28 to 42% and the frequency was 10 Hz.

Dynamic mechanical property (temperature sweep)

Dynamic mechanical property (temperature sweep) was determined by VA3000-type Dynamic Mechanical Thermal Analyzer (DMTA), produced by Rheometric Scientific, NJ. Testing conditions: the temperature was from -100°C to 100°C , the speed of temperature rise was $3^\circ\text{C}/\text{min}$, the frequency was 10 Hz, and the strain amplitude was 0.1% (operated in the tension mode).

Rolling power loss

Rolling power loss was determined by RPS-II Rubber Power Loss Testing Machine produced by Beijing Wanhui Yifang Technology Development, Beijing, China. The testing time was 30 min, the testing load was 30 kg and the testing rotational speed was 1200 r/min.

Surface and volume resistivity

Surface and volume resistivity was determined according to GB/T 1692-92 by PC68 Digital High Resistance Meter produced by The DC Instrument Branch to Shanghai Precision and Scientific Instrument, Shanghai, China, being conducted at 100 voltage and room temperature. The size of the square specimen is $12 \times 10 \times 2$ mm.

RESULTS AND DISCUSSION

Microstructure and morphology

The TEM photographs of SSBR filled with different ratios of SiO_2/CB are shown in Figure 2.

As shown clearly in Figure 2, the light part presents the rubber matrix, the transparent dark part is CB particles and the opaque dark part stands for SiO_2 . Figure 2(a1) shows CB particles with about 30 nm aggregate and form significant filler networks in the SSBR/CB composite, beneficial to conductivity; Figure 2(a4) displays that the 20–40 nm nano- SiO_2 particles are irregular and also easy to form filler networks in the SSBR/ SiO_2 composite, which is easy to accumulate static, because SiO_2 is insulator.

Figure 2(a2) shows that the irregular SiO_2 particles are inlaid into the CB networks in the composite with SiO_2/CB (20/50), and the filler dispersion is better than both SSBR/CB and SSBR/ SiO_2 composites; Figure 2(a3) shows SiO_2 and CB particles formed the interweaving networks in the composite

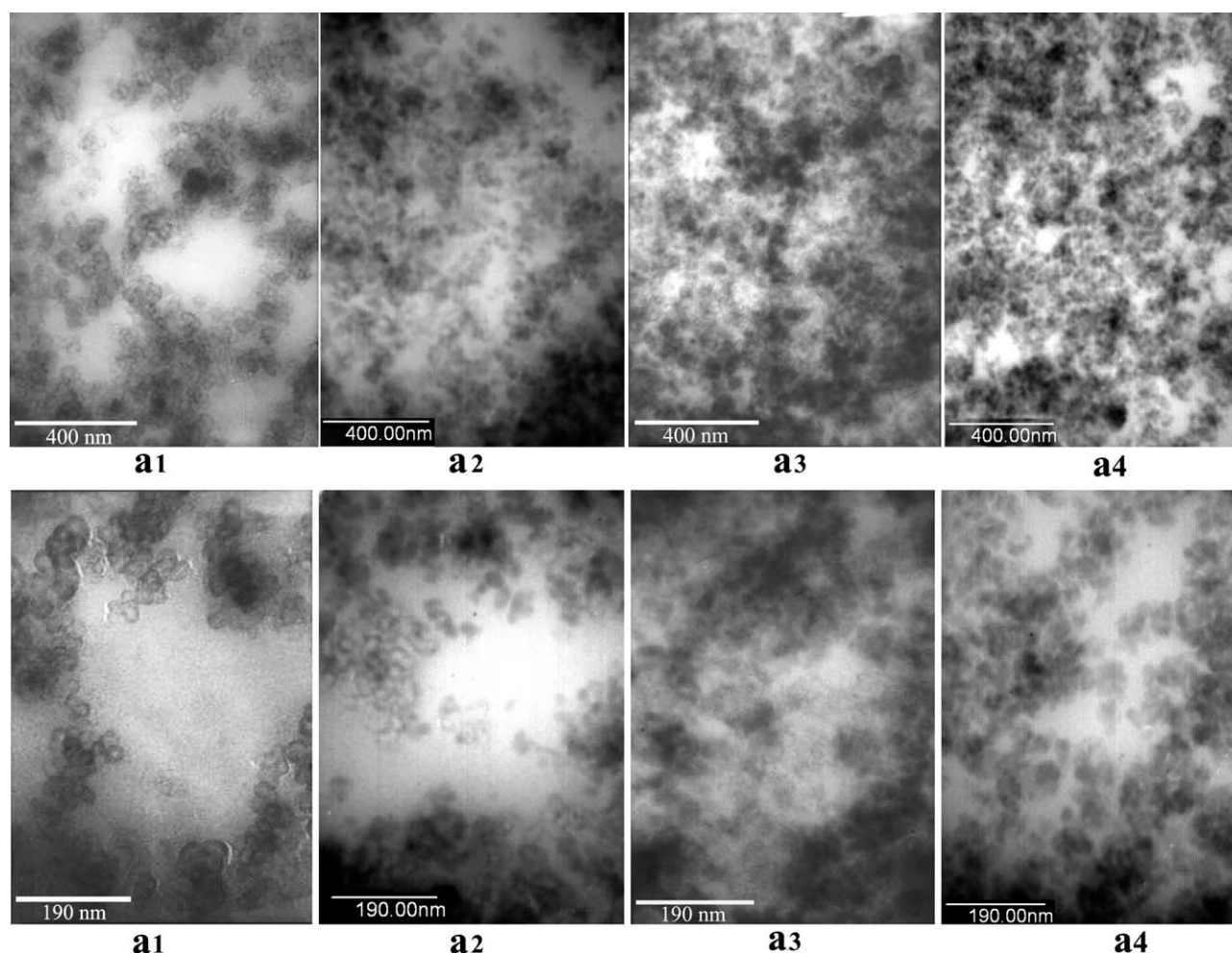


Figure 2 The TEM photographs of SSBR filled with different ratios of SiO₂/CB: (a1) 0/70, (a2) 20/50, (a3) 35/35, and (a4) 70/0.

with SiO₂/CB (35/35). CB particles are separated by SiO₂ for a certain distance, which will affect the anti-static property.

Vulcanizing and mechanical properties

Vulcanizing and mechanical properties of SSBR filled with different ratios of SiO₂/CB are shown in Table II.

As seen in Table II, the scorch time (t_{10}) of the rubber compounds changes little, while the optimum curing time (t_{90}) of those compounds increases with increasing amount of SiO₂. The curing rate of the compounds with SiO₂/CB (70/0) is obviously slow. Because there are many hydroxyl groups on the surface of SiO₂ powders, which can adsorb the vulcanization accelerator, resulting in the delayed vulcanization. The results indicate that it is feasible to increase the amount accelerator D properly with increasing amount of SiO₂.

Comparing with the SSBR/CB composite, the properties of the SSBR/SiO₂ composite are improved

in terms of modulus at 300%, tensile strength, elongation at break and tear strength. It is demonstrated that the comprehensive performances of SSBR/SiO₂ composites prepared by reaction blending are superior to those of the SSBR/CB composite. Furthermore, the mechanical properties of the composites with SiO₂/CB exhibit the "synergistic effect" compared with those of SSBR/SiO₂ or SSBR/CB composites. It indicates that when the rubber compound is mixed with SiO₂/CB dual phase fillers,^{17,18} the nano-scale dispersion will significantly weaken the networks formed by either CB or SiO₂ itself. Therefore, the fillers disperse well in the rubber matrix, and the interaction between fillers and rubbers is strong. Thus the mechanical properties and wear resistance of composites are improved.

In addition, the dynamic compression heat build-up and permanent set of SSBR composites filled with different ratios of SiO₂/CB decrease with the increasing amount of SiO₂ powder, and those of the SSBR/SiO₂ composite are the lowest. It shows that the strong bonding and self-lubricating effect of SiO₂

TABLE II
Vulcanizing and Mechanical Properties of the SSBR Composites Filled With Different Ratios of SiO₂/CB

Testing items	SiO ₂ /CB				
	0/70	20/50	35/35	50/20	70/0
t_{10} (min)	5.0	4.2	4.4	4.3	4.5
t_{90} (min)	8.3	8.2	9.2	11.0	17.1
Shore A hardness	68	68	65	65	64
Modulus at 300% (MPa)	11.6	18.3	15.6	15.1	12.8
Tensile strength (MPa)	17.3	22.8	22.2	21.5	20.3
Elongation at break (%)	356	378	409	454	402
Permanent set (%)	4	8	15	20	16
Tear strength (KNm ⁻¹)	46.6	48.1	57.9	50.7	46.9
Abrasion volume (cm ³ · 1.61 km ⁻¹) (Using a new and rough grinding wheel)	0.3695	0.2560	0.2783	0.2946	0.3576
Dynamic compression heat build-up (°C)	20.1	16.9	16.7	15.5	10.7
Dynamic compression permanent set (%)	3.20	2.84	2.76	2.64	2.40

powder could significantly reduce the friction loss between fillers and macromolecular chains. Moreover, it is the excellent filler dispersion that could decrease the compression permanent set of the composites. In summary, when the ratio of SiO₂/CB is 20/50, the comprehensive performances of the obtained composites reach a more excellent level.

Dynamic mechanical properties

$G' \sim \text{strain}$ and $\tan \delta \sim \text{strain}$ curves of SSBR filled with different ratios of SiO₂/CB (i.e., strain-sweep spectra) are shown in Figure 3.

Figure 3(a) shows that $\Delta G'$ (the decreasing amplitude of G' from 0.28 to 42% of the strain) of the SSBR/CB composite is the largest (i.e., the higher the Payne effect¹⁹⁻²¹ is, the worse the dispersion of fillers is). $\Delta G'$ of the SSBR/SiO₂ composite is smaller than that of the SSBR/CB composite, whereas somewhat larger than that of the composites with SiO₂/CB, which indicates that either of SSBR with the two kinds of nano-fillers

in large filler content presents poor dispersion (i.e., with aggregates of the filler particles). Because the more the breakup of the aggregates are in shearing action, the higher the $\Delta G'$ value is. It could also be proved by TEM photograph. Each of the composites filled with SiO₂/CB shows low Payne effect and good filler dispersion (i.e., the fillers disperse in the nano-scale level with few aggregates). Especially when the ratio of SiO₂/CB is 20/50, Payne effect is the lowest. Because When a large number of CB are mixed into the SSBR matrix having been mixed with a small amount of SiO₂ beforehand, the viscosity of the compounds is rapidly increasing, which makes the fillers suffer stronger shearing action in the force field. Besides, the compatibility between CB and SSBR is excellent. Thus the nano-scale dispersion is enhanced. This shows that the composite filled with an appropriate ratio of SiO₂/CB dual phase filler has good filler dispersion.

Figure 3(b) shows that, when the strain is less than 10%, $\tan \delta$ value of the SSBR/SiO₂ composite

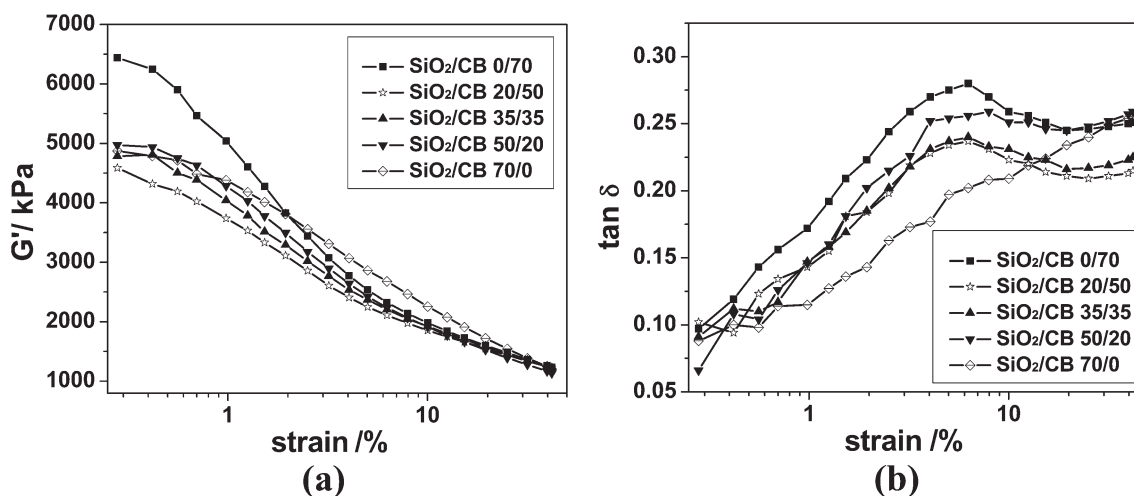


Figure 3 $G' \sim \text{strain}$ and $\tan \delta \sim \text{strain}$ curves of SSBR filled with different ratios of SiO₂/CB.

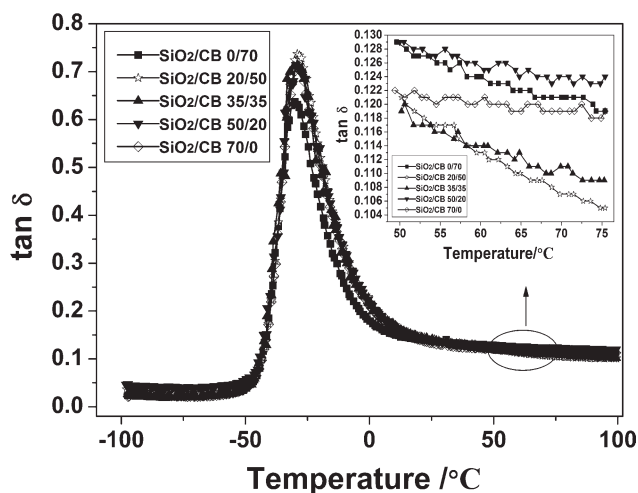


Figure 4 Tan δ versus Temperature curves of SSBR filled with different ratios of SiO₂/CB.

increases with increasing strain and is the smallest; When the strain is larger than 10%, tan δ value of the SSBR/SiO₂ composite increases sharply with increasing strain, close to the tan δ value of the SSBR/CB composite. This is because in spite of bonding with macromolecular chains, SiO₂ powder modified by a silane coupling agent, forms obvious filler networks in the rubber matrix. When the external forces are strong enough to destroy the filler networks, the interfacial friction between SiO₂ particles and the rubber matrix increases sharply, resulting in increasing tan δ of the composite. Tan δ values of the composites filled with SiO₂/CB are between those of the SSBR/CB and SSBR/SiO₂ composite in the range of the strain from 0.28% to 10%, among which tan δ value of the composite with SiO₂/CB (20/50) is the smallest, because the dispersion of fillers is good and the breakup of the filler aggregates is less. Furthermore, there is apparently no sharp increase in tan δ values of the three SSBR/SiO₂/CB composites under the large strain. It is suggested that the rubber compound with the appropriate ratio of SiO₂/CB dual phase fillers, could remarkably reduce filler networks and decrease the rolling resistance, owing to the higher polymer-filler interaction and lower filler-filler interaction endowed by reaction blending and the silane coupling agent.²²

According to the Time-Temperature Equivalence Principle²³ and polymer viscoelastic theory, an ideal

material which is able to meet the requirements of green tires should give a low tan δ value at a temperature (60°C) to reduce rolling resistance. The ideal material should also demonstrate a high tan δ value at lower temperatures (0°C) to obtain high wet-skid resistance.^{24,25} The temperature-sweep curves determined by DMTA and their characteristic values of SSBR composites filled with different ratios of SiO₂/CB (i.e., temperature-sweep spectra) are shown in Figure 4 and Table III.

As is depicted in Figure 4 and Table III, the addition of SiO₂ powder could make the T_g peaks of SSBR composites move right and become wider. Compared with the SSBR/CB composite, tan δ of the SSBR/SiO₂ or SSBR/SiO₂/CB composites at 0°C increases while tan δ at 60°C slightly decreases or doesn't change so much. Among these, tan δ of the composite with SiO₂/CB (20/50) at 0°C is the largest and at 60°C is the smallest, which demonstrates the composite has better wet-skid resistance and lower rolling resistance as well as higher wear resistance. Therefore, the "magic-triangle" properties of the composite used for the tire tread are well balanced.

The rolling power loss

The rolling power loss is to simulate the energy loss of the tires moving on the road surface with the mechanical energy converted into heat. The dynamic deformation, heat generation and power loss values of the composites were shown in Table IV.

Table IV shows that the rolling power loss values of SSBR/SiO₂ or SSBR/SiO₂/CB composites are lower than those of the SSBR/CB composite, and the rolling power loss value of the composite with SiO₂/CB (20/50) is the lowest, which is in accordance with the trend of tan δ in the strain range from 15 to 30% in Figure 3(b), i.e., the larger the tan δ value is, the higher the rolling power loss will be. Although the SSBR/SiO₂ composite displays large dynamic deformation, the power loss value and heat generation of this composite are less than those of the SSBR/CB composite, which indicates that adding nano-SiO₂ to the SSBR composite could remarkably reduce hysteresis loss of the composite. In addition, the composite with low rolling power loss and heat generation has high wear resistance,²⁶ as can be seen from Table II.

TABLE III
 T_g and Tan δ Values of SSBR Filled with Different Ratios of SiO₂/CB

Testing items	SiO ₂ /CB					Standard deviation
	0/70	20/50	35/35	50/20	70/0	
T_g (°C)	-30.1	-29.1	-30.5	-29.6	-28.0	0.971
Tan δ (0°C)	0.18	0.23	0.22	0.22	0.21	0.019
Tan δ (60°C)	0.12	0.11	0.11	0.13	0.12	0.008

TABLE IV
Power Loss in the Rolling Process of SSBR Filled With Different Ratios of SiO₂/CB

Testing items	SiO ₂ /CB				
	0/70	20/50	35/35	50/20	70/0
Dynamic deformation (%)	18.0	17.8	18.0	29.4	23.9
Heat generation (°C)	79.9	66.1	66.0	74.4	70.7
Power loss value (J/r)	4.26	2.62	2.97	3.70	3.23

It could be seen from the experimental results that the SSBR composite filled with SiO₂/CB (20/50) shows good filler dispersion, low rolling resistance, high wet-skid and wear resistance.

Electrical properties

Surface and volume resistivity of SSBR filled with SiO₂/CB is given in Figure 5.

As seen from Figure 5, the surface and volume resistivity of the SSBR/CB composite is the lowest, which indicates that there are many conductive networks formed by CB in the SSBR/CB composite. This could also be proved by the analysis results from the dynamic mechanical properties and morphology of the composite. The surface and volume resistivity of the composites filled with SiO₂/CB less than 35/35 (i.e., from 0/70 to 35/35), exhibit a gradual rise with the increasing amount of SiO₂ and increase one order of magnitude with increasing 15–20 phr SiO₂. The surface and volume resistivity of the composites filled with SiO₂/CB over 35/35 (i.e., from 35/35 to 70/0), increase 5–6 orders of magnitude with increasing 1520 phr SiO₂.

Due to the nano-scale dispersion of SiO₂ and CB in the SSBR/SiO₂/CB composites, CB particles are separated by SiO₂ for a certain distance, and the conductivity decreases. According to the tunneling conductivity theory,^{27,28} when the isolation distance

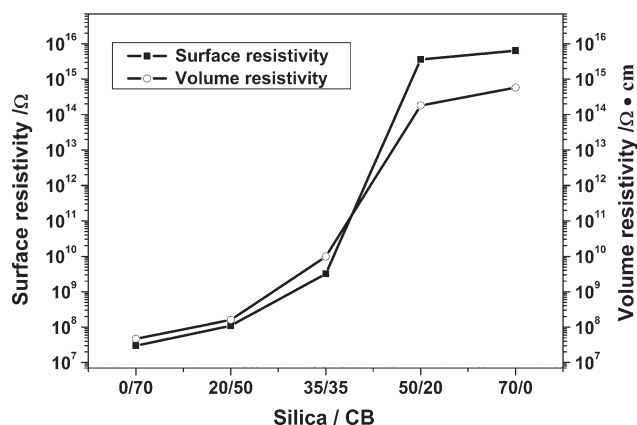


Figure 5 The surface and volume resistivity of SSBR filled with different ratios of SiO₂/CB.

between CB particles is less than 10 nm or could be shortened to 10 nm by the external forces, electron transition would still occur in this state and the composite displays a certain antistatic property; when the isolation distance is so long that the external forces could hardly make it less than 10 nm, the isolator feature is displayed.

According to ASTM D991, the antistatic rubber products are considered to have a resistance of 10⁴ to 10⁸ Ω. Accordingly, the antistatic range is from 10⁶ to 10¹⁰ Ω·cm after the unit conversion from resistance to resistivity. Thus, the ratio of SiO₂/CB should be less than 1/1 (i.e., from 35/35 to 0/70), and for the SSBR composites with 70 phr fillers, the ratio of SiO₂/CB in 35/35 was the percolation threshold of the antistatic property.

CONCLUSIONS

1. CB particles in the SSBR/CB composite present 30 nm spherical particles and form filler networks. The SSBR/CB composite shows large Payne effect, high internal friction and excellent antistatic property; SiO₂ powder in the SSBR/SiO₂ composite presents 20–40 nm irregular particles and also forms filler networks. The SSBR/SiO₂ composite displays low internal friction and large static accumulation.
2. The mechanical properties of SSBR composites filled with SiO₂/CB exhibit the “synergistic effect.” Among these, the composite with SiO₂/CB (20/50) shows good filler dispersion with low heat generation and the “magic-triangle” properties are well balanced.
3. For the SSBR composites with 70 phr fillers, the ratio of SiO₂/CB in 35/35 is the percolation threshold for the antistatic property. To satisfy the request of the antistatic property, the ratio of SiO₂/CB should be less than 35/35.

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