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Research Article

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Posted Date: November 13th, 2026

DOI: https://doi.org/10.21203/rs.3.rs-11061750/v1

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Additional Declarations: No competing interests reported.

Effect of Carbon Black Morphology on Electrical and Mechanical Properties of Filled Natural Rubber

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ABSTRACT

The balance between electrical conductivity and mechanical reinforcement is a critical challenge in the development of conductive rubber composites for aircraft tire applications. In this work, the electrical and mechanical behaviors of natural rubber (NR) composites filled with a conventional reinforcing carbon black (N234) and a high-surface-area conductive carbon black were systematically investigated. Compared with N234-filled NR, the conductive carbon black composite exhibits significantly lower volume resistivity and improved stability of electrical conductivity under cyclic tensile deformation, which is attributed to the formation of a more continuous and damage-resistant conductive network. These results demonstrate that electrical performance is primarily governed by the integrity of the carbon black network, while mechanical behavior is dominated by polymer-filler interfacial reinforcement. This work provides new insight into the independent evolution of conductive and reinforcing networks and offers guidance for designing balanced conductive rubber systems for tire applications.

Keywords: conductive carbon black, reinforcing carbon black, specific surface area, filled rubber composites, electrical properties, stress softening

1. Introduction

Static electricity is generated by friction between aircraft tires and the ground during takeoff and loading, as well as by the friction of an airplane with various environmental conditions like airborne particles and charged clouds. Static electricity can lead to unstable operation of electrical equipment, and even can fail the electronic components on the airplane. It also easily triggers fire hazards during refueling and poses a serious threat to the physical health of crew and passengers [1, 2]. Therefore, the aircraft tires are required to be made of conductive rubber, which permits electricity to flow through it with minimal resistance and expels electricity to the ground in time. Natural rubber (NR) filled with carbon black are widely used for aircraft tire tread application with good strength, tear resistance and excellent antistatic properties [3]. It is well known that carbon black plays a dominant role in the mechanical and electrical properties of filled-rubber composites. The influence of carbon black on the mechanical properties of filled rubber is complicated, and can be summarized by a combination of the following mechanisms such as strain amplification, interfacial interaction between filler and polymer, occlusion of rubber and filler agglomeration [4-9].

Whenever such materials are used in aircraft tires, they are subjected to hundreds of times of high temperature, high pressure, high-speed friction and extreme temperature changes. These working conditions induce wear and fatigue damage, destroy the carbon black network, and further result in the degradation of electrical conductivity [10, 11]. Therefore, improving the electrical conductivity of rubber by incorporating various materials has long been a major research focus. Barbosa and

coworks [12] reported that the electrical conductivity of NR composites filled with carbon nanotube (CNT) increased remarkably when the CNT content reached 2.5 wt%. A comprehensive study on NR composites reinforced with synthetic graphite and chitosan was conducted by Kitsawat et al [13]. Compared with neat NR films, NR composite films filled with 60 phr graphite exhibit a remarkable improvement in electrical conductivity. Pazilova et al [14] investigated four formulations of carbon black-filled butadiene methylstyrene rubber. The selected formulation exhibited improved electrical conductivity, tensile strength and tear resistance. Nevertheless, most of the relevant research concentrates on improving electrical conductivity. Far less attention is paid to the mechanical properties, as well as the relationship between electrical properties and mechanical properties.

Herein, conductive carbon black was adopted to improve the electrical conductivity of natural rubber compounds and the effect of cyclic strain on the electrical and mechanical properties of the composites was investigated, compared with those of the reinforcing carbon black (N234) commonly used in aircraft tires. This work integrates the mechanical and electrical properties of the two compounds, and analyzes the mechanisms underlying the distinct effects of fillers on their mechanical and electrical properties, aiming to provide theoretical guidance for the formulation development of highly conductive tread rubber for aircraft tires.

2. Experiment

2.1 Materials and Instruments

NR (STR20) was obtained from Thai Hua Rubber Public Co., Ltd., Thailand.

N234 (CB1) was obtained from Shanghai Cabot Chemical Co., Ltd., China. Conductive carbon black (CB2) was supplied by Solprima Carbon Material Co., Ltd., China. Zinc oxide (ZnO) was obtained from Weifang Longda Zinc Industry Co., Ltd., China. Steric acid (SA) was purchased from Lianyungang Yihai Petrochemical Industry Co., Ltd., China. Anti-ozone wax was obtained by Shandong Yanggu Huatai Chemical Co., Ltd., China. Poly(1,2-dihydro-2,2,4-trimethyl-quinoline) (RD) was bought from Shandong Stair Chemical Co., Ltd., China. N-1,3-dimethylbutyl-N'-phenyl-p-phenylenediamine (4020) was supplied by Sennics Co., Ltd., China. Accelerator Ncyclohexylbenzothiazole-2-sulfonamide (NS) was obtained from Shandong Sunsine Chemical Co., Ltd., China. Sulfur purchased from Linyi Xin'an Chemical Co., Ltd., China. All the materials were used as received.

2.2 Preparation of the composites

The formulations used in this study are shown in Table 1. All compounds were mixed in an internal mixer (1 L) equipped with tangential rotors at a constant rotor speed of 80 rpm in the first mixing stage. The rubber was masticated for 30 s then fillers, zinc oxide, stearic acid and the antioxidants were added. In a second mixing stage sulfur and accelerators were added at a rotor speed of 55 rpm. The compounds were cured at 150℃ for an optimum cure time pre-determined from the oscillating rheometer study (ODR 2000 of Alpha Technologies, USA).

Table 1 Formulation of Compounds (unit: phr)

Ingredients	CB1	CB2
STR20	100	100
CB1	50	
CB2		50
SA	2	2
ZnO	3.5	3.5
Wax	1	1
RD	1.5	1.5
4020	2	2
NS	1.2	1.2
Sulfur	1.0	1.0

2.3 Measurement of surface energy of carbon black

Inverse gas chromatography (IGC) was used to measure the surface energy of carbon black according to literature methods [15]. Tests under infinite dilution were carried out on the Agilent Technologies 7890B GC System fitted with a flame ionization detector (FID). A 5 cm × 2 mm stainless steel column was filled with around 0.1 g carbon black. Helium served as the carrier gas at a flow rate of 20 cm^3/min. The column was pre-conditioned at 180 °C for 20 h and measurement was conducted at 180 °C. To satisfy the condition of adsorption at infinite dilution corresponding to zero coverage and conditions of gas chromatography linearity, the injection volume of probe vapor was controlled below 0.1 μL. *n*-Butane, *n*-pentane, *n*-hexane and *n*-heptane were used as probes.

2.4 Measurement of the mechanical properties

The tensile properties of the samples were evaluated using a universal testing machine (Instron Corporation) with dumbbell specimens cut from 2 mm thick sheets in accordance with GB/T 528-2009. Other mechanical properties were tested following the corresponding national standard of China.

2.5 Measurement of the electrical properties

The electrical resistivity of the specimens was measured using a UNI-T digital multi meter (UT61E). The measurement of the electrical resistivity in tension was carried out with a dumbbell type specimen. The dumbbell specimen having total length 115 mm, distance between grips 80 mm, width 4 mm and thickness 2 mm was cut from the cured rubber sheet with gage length of 20 mm. Before measuring each specimen, sample surface was cleaned with ethanol to remove any traces of impurities. To measure the electrical resistivity at different strains, the sample was clamped to the Instron Universal Testing Machine (UTM 5969), and attached with the crocodile clips of the multi-meter at a separation of 20 mm. The samples were stretched stepwise to extension ratios of 1.25, 1.5, 1.75, 2, 3, 3.5 and 4.0, and then sequentially retracted to the corresponding extension ratios, where the cross-head speed was 75 mm/min. All data readings were made at about 10 s after each elongation step to standardize the effect of stress relaxation.

The electrical resistivity (ρ) was calculated as follows:

$$\rho = \frac{RA}{l}$$

Where R is the measured resistance value, A is the instantaneous cross-sectional area, equal to A_0/λ, where A_0 is the initial cross-sectional area and λ is the tensile extension ratio in the test piece; l is the fixed distance between the crocodile clamps on the sample.

2.6 Measurement of the stress softening

The test conditions are the same as those for electrical resistivity. The specimens were tested at room temperature on the Instron Universal Testing Machine (UTM 5969)

at a stretching rate of 75 mm/min. Each sample was directly stretched to the extension ratio of 4.0 and then retracted to zero stress, and this cycle was repeated 10 times in total.

2.7 Condition of recovery experiment

The specimens after cyclic stretching were placed at room temperature for 24 h of recovery, and the subsequent tensile test conditions were the same as described above.

3. Results and discussion

3.1 The mechanical and electrical properties of the two compounds

The mechanical and electrical properties of the two compounds are shown in Table 2, and the analytical properties of the relevant carbon blacks are given in Table 3. It is observed that the 300% modulus of the CB2-filled rubber composite is significantly lower than that of the CB1-filled one. This may be attributed to the excessively high specific surface area and high surface energy of CB2. Such a high specific surface area and high surface energy makes it difficult to be dispersed in the rubber matrix, greatly reduces the effective reinforcing surface area, and consequently leads to weak polymer-filler interaction, which lowers the 300% modulus. Furthermore, the weaker interaction between CB2 and the rubber matrix makes polymer chains easily slip and detach from the filler surface during extension, thereby inducing void formation. The growth of voids easily relieves excessive stress in the localized area, causing the load to be more equally distributed [9, 16]. This may be beneficial to improve elongation at break of CB2-filled rubber composite. Moreover, poor dispersion of CB2 in the rubber matrix increases microscopic defects and stress concentration sites of the composites, resulting

in a reduction in the tensile strength and tear strength.

Table 2 Physical characteristics of CB1- and CB2-filled Vulcanizates

Properties	CB1	CB2
Hardness, RT, Shore A	60	63
Tensile strength, MPa	30.9	28.1
Modulus at 100% elongation, MPa	2.8	1.9
Modulus at 300% elongation, MPa	15.1	8.2
Elongation at break, %	560	645
Tear strength, N/mm	119	102
Volume resistivity, Ω*cm	1.3E+3	2.0E+1

Table 3 Analytical properties of relevant carbon blacks

Ingredients	CB1	CB2
Nitrogen surface area (m^2/g)	114	400
Statistical thickness surface area (m^2/g)	111	259
DBP absorption number ($cm^3/100$ g)	125	136
CDBP absorption number ($cm^3/100$ g)	102	104
Surface energy γ_s^d (mJ/m^2)	294	405

In addition, it can be observed that CB2 has a much lower electrical resistivity than CB1. Before explaining this phenomenon, it is necessary to understand the conductive mechanism of carbon black-filled rubber composites. The conductive mechanism involves two main aspects: Ohmic conductance and the tunneling effect [17, 18]. Several parameters affect the overall conductivity of composite materials, including filler content, types, morphology and dispersion quality [19]. The filler content is particularly dominant in determining the formation of the conductive network. The superior conductivity of composites can only be achieved when the filler content is above a certain ratio, known as the percolation threshold [20-22]. At this state, the formation of continuous conductive routes or networks enables electron flow throughout the materials by following Ohmic conductance. When filler content is near or below the percolation threshold, it is hard to form direct contact of conductive fillers.

Instead, the narrow gap or short distance at nm level between adjacent fillers is formed, and the electrons can go through the void from one conductive particle to another by the tunneling effect [23]. For the two materials investigated in this paper, the filler content is relatively high, so it can be considered that Ohmic conductance plays a dominant role [24, 25].

Therefore, the lower electrical resistivity of CB2 is attributed to its ability to form a strong conductive network which is closely related to its higher specific surface area. Based on the above conductive mechanism, the smaller distance between carbon black particles facilitates electron transport and promotes the formation of conductive network. It is known that the most important factor governing the distance between aggregates is the specific surface area of the filler [26]. Therefore, the larger specific surface area of CB2 results in the closer distance between particles, making it easier to form a strong conductive network. Meanwhile, some research indicates that the effect of filler structure on electrical resistivity is weaker than that of specific surface area [27]. Since CB1 and CB2 have comparable structure (Table 2), the effect of filler structure is not considered here.

Furthermore, differences in the shape of aggregates are identified between the two carbon blacks, as shown in the transmission electron microscopy (TEM) images in Figure 1. It is observed that the primary particle size of CB1 is 18~21 nm, while that of CB2 is 12~15 nm. Apparently, CB2 has smaller primary particles than CB1, which also accounts for the shorter distance between CB2 aggregates. Moreover, the aggregates of CB2 seem to be more interconnected which implies that electron transmit may occur

more easily.

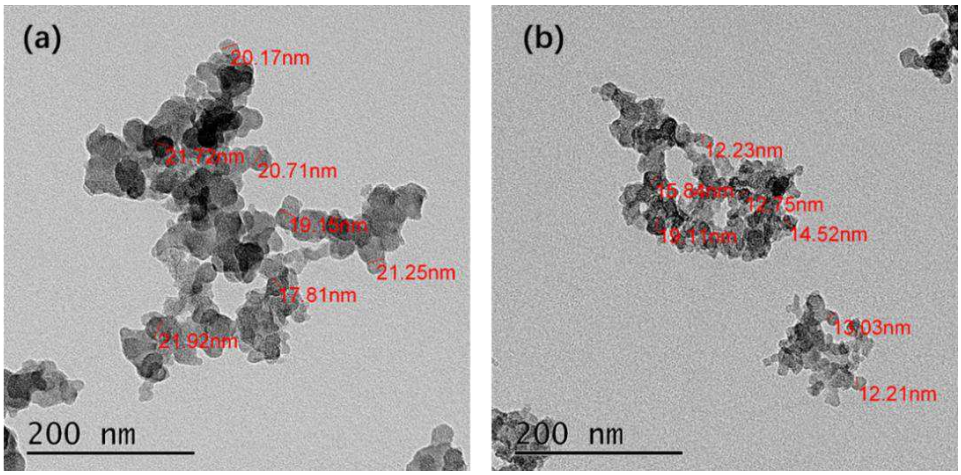


Fig. 1 TEM images of carbon black (a) CB1 and (b) CB2

3.2 The variation of electrical properties in the first stress-strain cycle

Considering that aircraft tires are subjected to repeated stress and strain, it becomes necessary to understand how electrical and mechanical properties change with repeated stress-strain cycles. Figure 2 shows a comparison of the electrical resistivity of CB1- and CB2-filled compounds in their first stress-strain cycle. It is found that the electrical resistivity of CB1 increases with the increasing of strain, followed by a decrease, and then attains a plateau value. The initial increase in electrical resistivity at low strain (λ=1.25~1.5) has been attributed to the breakdown of the carbon black aggregates and agglomerates, which is thought to result in a reduction of conductive paths [28-31]. The speculation was confirmed by scanning electron microscope (SEM) images of CB1 specimen under tension in Figure 3. Figure 3a shows the SEM image of the specimen before stretching, in which the carbon black aggregates are randomly and disorderly dispersed in the rubber matrix. As shown in Figure 3b (λ = 1.5), there are some voids around the carbon black generated by the breakdown of carbon black aggregates and agglomerates, resulting in fewer conductive paths.

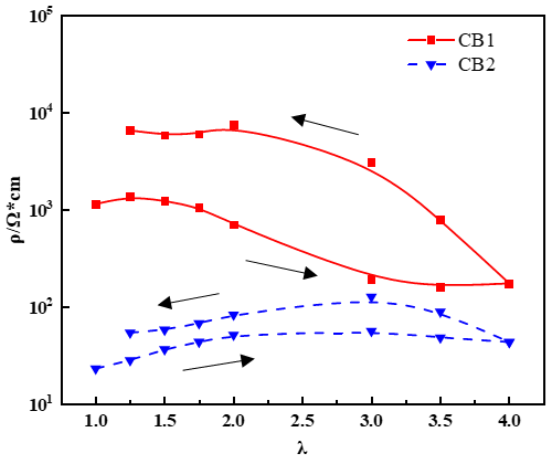


Fig. 2 Electrical resistivity versus extension ratio data for the first stress-strain cycle for CB1- and CB2-filled vulcanizates

Subsequent reduction in the electrical resistivity has been presumed to result from the orientation of the carbon black aggregates in the direction of strain [29, 30]. The orientation effect leads to the reduction in the distance between carbon black aggregates in the equatorial direction [32]. This could generate new conductive paths which would result in a reduction of electrical resistivity. As show in Figure 3c, the carbon black aggregates appear to be dispersed along the stretching direction, which may prove the orientation of carbon black aggregates to some extent. Notably, although the breakdown of carbon black aggregates and agglomerates still occurs at this stage, the orientation of carbon black aggregates dominates the process, ultimately leading to a reduction in electrical resistivity.

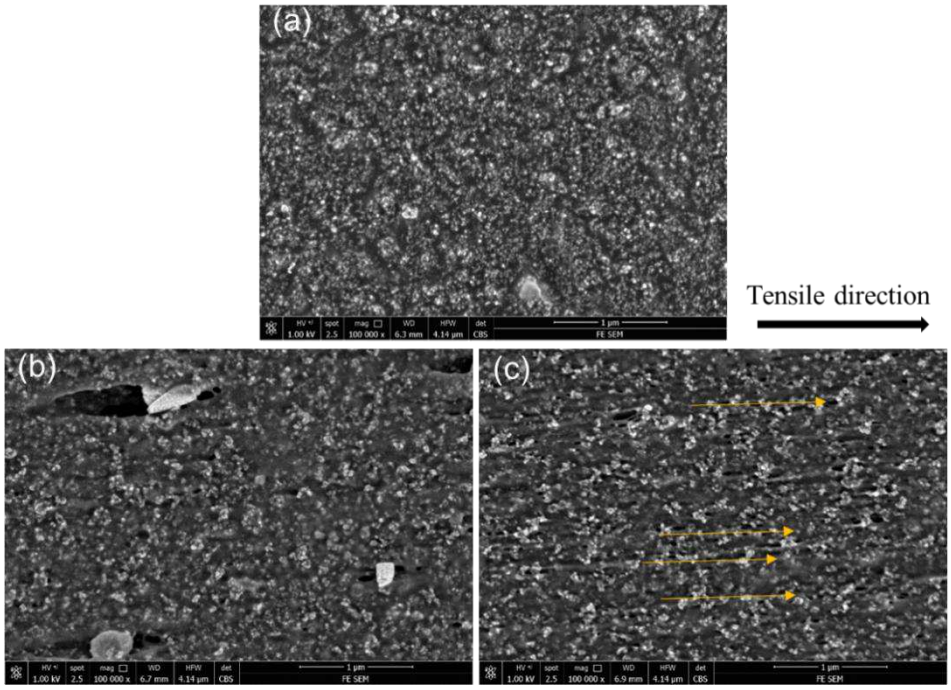


Fig. 3 SEM images of CB1-filled rubber composite with extension ratio (a) λ=1 and (b) λ=1.5 and (c) λ=2.5

Eventually, the curve tends to level off, indicating that the carbon black networks may no longer change significantly at this strain. At this stage, most structural changes may occur within the rubber network and at the rubber-filler interface, such as the slippage and the detachment of polymer chains on the filler surface, which appear to have little effect on the electrical resistivity of the material.

In contrast to CB1, the electrical resistivity of CB2 initially increases and then gradually levels off with increasing strain, and its resistivity exhibits a smaller variation amplitude with tensile strain. At small to medium strains (λ=1.0~2.0), the breakdown of the carbon black aggregates and agglomerates causes the increase of electrical resistivity. Due to its robust and damage-resistant conductive network, CB2 shows much lower electrical resistivity and only minor variations in electrical resistivity upon tensile strain, meanwhile, the decreasing of electrical resistivity induced by carbon black aggregate orientation is hardly observed. Accordingly, its resistivity becomes

stable at extension ratio above 2.0, in contrast to the decreasing trend observed for CB1 at extension ratio from 1.25 to 3.0. During retraction, CB1 and CB2 follow similar variation trends. The electrical resistivity rises steadily up to a given strain, then declines and gradually levels off. Moreover, the electrical resistivity during retraction is higher than that observed during corresponding initial elongation. The results may be explained as follows. Two main phenomena occur during retraction: (1) recovery caused by the movement of rubber molecular chain segments; and (2) reformation of new conductive networks. At the initial stage of retraction, the orientation of carbon black aggregates may be gradually disrupted. The filler aggregates rearrange but fail to reform an effective conductive network, leading to an increase in resistivity. As further retraction occurs, the retraction and adjustment of rubber molecular segments may reduce the distance between carbon black particles, facilitating the formation of new conductive networks and thus lowering the resistivity. Ultimately, with the reformation of the new conductive networks nearly complete, resistivity remains steady.

Notably, CB2 shows an earlier inflection point (λ=3.0), which implies that its conductive network reforms faster than CB1 (λ=2.0). Meanwhile, the electrical resistivity hysteresis loop of CB2 is smaller than that of CB1 throughout the stress-strain cycle. This demonstrates that the conductive networks of CB2 are more stable and less likely to be damaged under stress and strain, with smaller fluctuations in resistivity. In view of its electrical properties, CB2 could be applied to improve the stability of electrical conductivity for aircraft tire treads.

3.3 The variation of stress in the first stress-strain cycle

Apart from the electrical behavior, cyclic stress softening tests were also carried out, and the first stress-strain curve of the two compounds are presented in Figure 4. As observed, the modulus upon retraction is lower than that during tensile loading. This phenomenon can be attributed to the damage to the polymer network and filler-polymer interfaces, along with irreversible energy dissipation caused by internal friction of rubber molecular chains during deformation [33]. The amount of mechanical hysteresis of CB2 was significantly smaller than CB1. This may be attributed to the poor dispersion of CB2 and the weak polymer-filler interaction of the CB2-filled composite discussed above. On the one hand, CB2 exhibits poor dispersion and severe filler agglomeration in the rubber matrix, which reduces the effective interfacial area for reinforcement. Therefore, the energy loss originating from the motion of rubber molecules at the rubber-filler interface is low, such as desorption and the slippage of rubber molecules on the filler surface. On the other hand, the weak interaction between CB2 and the rubber leads to low interfacial bonding strength, namely a thinner rubber shell [9]. Only a limited number of polymer chains adjacent to the filler surface are restricted in mobility [7, 34], while most of the polymer chains can move relatively freely, leading to further decreased energy dissipation.

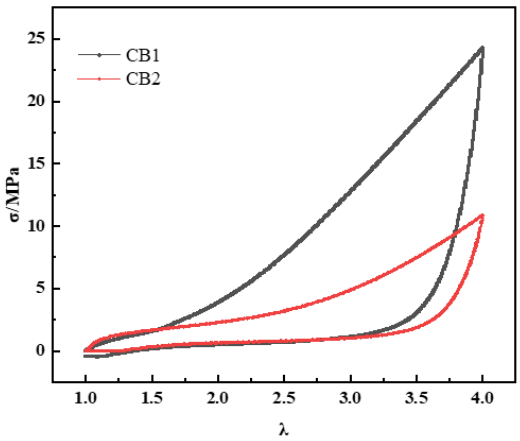


Fig. 4 The first stress-strain cycle for CB1- and CB2-filled vulcanizates

3.4 The cyclic stress softening of the two compounds

Figure 5 shows the cyclic stress softening curves of both specimens. The typical Mullins effect can be clearly observed, by which the mechanical hysteresis is significantly larger for the first loading cycle and stress softening occurs during the second cycle [35-37]. Moreover, the modulus of both composites partially recovered after 24 h of storage. This implies that the rubber molecular chains may undergo re-adsorption at the filler surface and re-entanglement, and thus the reinforcement-related elastic networks are partially re-established [38]. These elastic networks include rubber networks, rubber-filler interfacial structures and filler networks.

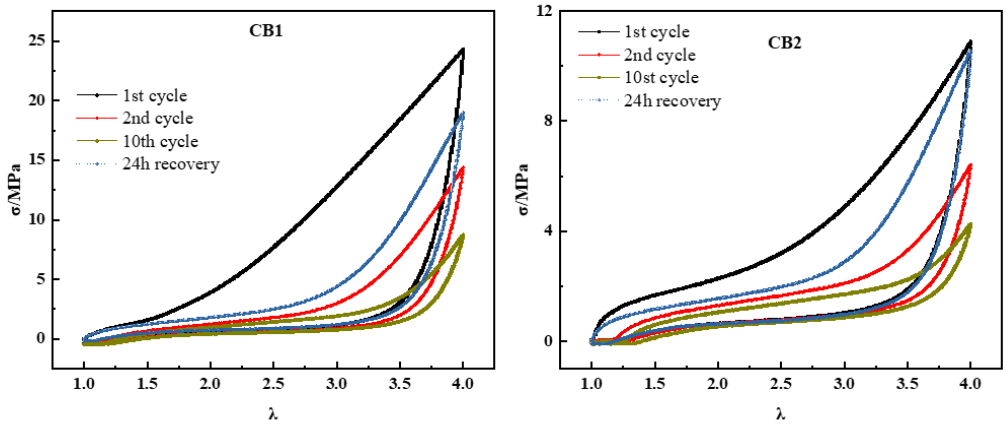


Fig. 5 Stress-strain curves of CB1- and CB2-filled vulcanizates

The stress softening degree is illustrated in Figure 6. The stress softening degree (ΔW) is characterized by the percentage reduction in strain energy caused by stretching to a given extension ratio:

$$\Delta W = (W_1 - W_2) / W_1 \times 100\%$$

Where W_1 is the energy required for the first stretching of the specimen to a given extension ratio. This energy was calculated from the raw data of the first stress-strain curve at specific extension ratios of 1.25, 1.5, 1.75, 2, 2.5, 3.0, 3.5 and 4.0. And W_2 is the energy required for the second stretching to the same strain, obtained using the same calculation method as W_1.

In the case of both materials, ΔW decreases with increasing strain, undergoes a minimum at λ about 1.8, and then increases with strain. It is known that the softening effect at low strain is mainly related to the breakdown of filler agglomerates. With increasing strain, the number of broken-down agglomerates is reduced, and so ΔW decreases. At the same time, other processes may take place such as the breakage of polymer chains, or their detachment to adjacent filler particles, and the movement of the aggregates through the surrounding matrix. Due to the viscoelastic nature of rubber, some of these processes are reversible after sufficient relaxation, and some are irreversible. Therefore, during the second and the subsequent stretching cycles, stress softening occurs. The increase of strain may promote these processes, which is responsible for the increase of ΔW with strain.

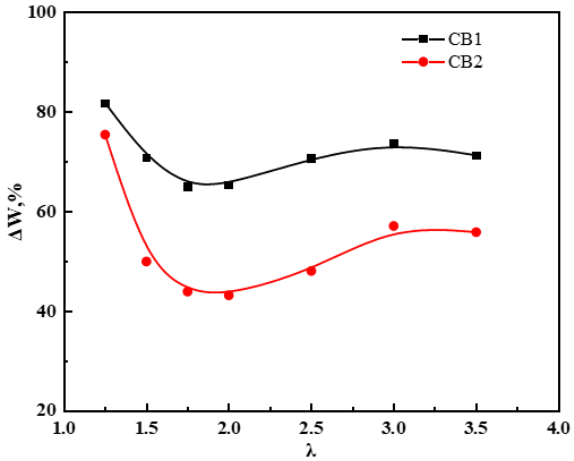


Fig. 6 The stress softening degree of the CB1- and CB2-filled vulcanizates

CB2-filled rubber composite exhibits a lower degree of stress softening at small strains compared with CB1. This is probably due to the excessively high specific surface area and high surface energy of CB2, which makes it difficult to be dispersed in rubber matrix. As a result, numerous aggregates show negligible interaction with the rubber. The CB2-filled rubber composite contains more filler agglomerates than the CB1 counterpart, while its filler network is perhaps relatively weaker. Consequently, the filler network of CB2 is easily disrupted at small strains, leading to a low degree of stress softening. At large strain, the lower stress softening degree of the CB2-filled rubber composite may be attributed to the poor polymer-filler interaction as mentioned above.

3.5 The variation of electrical properties under stress-strain cycles

The corresponding electrical properties under stress-strain cycles are shown in Figure 7. Consistent with the variation in mechanical properties, significant carbon black network breakdown occurs in the first cycle, while less difference is seen between the second and the tenth cycles. Likewise, as the number of cycle increases, CB2 exhibits a smaller resistivity variation and more stable electrical behavior, in agreement

with its strong conductive networks due to the higher specific surface area discussed earlier.

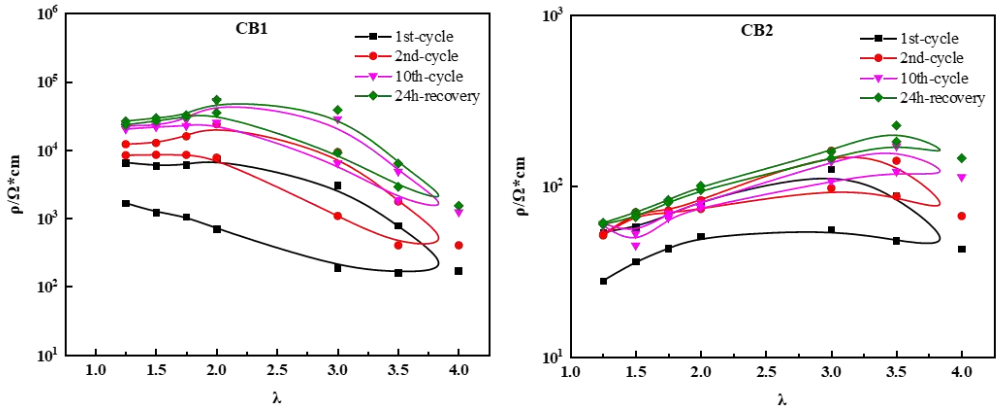


Fig. 7 Electrical resistivity versus the extension ratio during cyclic tensile tests for CB1- and CB2-filled vulcanizates

After 24 h of storage, the electrical resistivity can hardly recover which means that the carbon black agglomerates contributing to the electrical conductivity suffered irreversible damage and could not be reformed by relaxation. This phenomenon seems inconsistent with the above changes in mechanical properties, since the relaxation process enables partial re-establishment of the reinforcement-related elastic networks, such as the re-adsorption of rubber molecule chains at the filler surface and the re-entanglement of polymer chains. It is demonstrated that carbon black network breakdown has a considerably greater effect on electrical resistivity, and the reinforcement-related rubber network plays a major role in mechanical behavior.

4. Conclusions

To meet the requirements for electrical conductivity and mechanical properties of aircraft tire tread, two types of carbon black filled rubber composites were investigated in this work. Compared with normal reinforcing carbon black, conductive carbon black delivers lower and more stable electrical resistivity, benefiting from its high specific

surface area that forms a strong and damage-resistant conductive network. However, excessively high specific surface area leads to poor dispersion and weak reinforcement in the rubber matrix, resulting in a significant decrease in 300% modulus. Mixing conductive and reinforcing carbon black is therefore proposed to balance tire performance, representing a valuable direction for future research. Cyclic tensile and resting recovery tests demonstrate that electrical resistivity is largely irreversible, as the conductive network could not be reformed by relaxation. In comparison, partial modulus recovery is achieved due to the reformation of reinforcement-related elastic networks. The results further verify that the breakdown of carbon black aggregates has a far greater impact on electrical resistivity, whereas mechanical behavior is mainly determined by the reinforcement-associated rubber network.

Acknowledgements

This work was supported by the National Natural Science Foundation of China (51973102); Qingdao Natural Science Foundation (24-4-4-zrjj-193-jch). Qingdao Science and Technology Benefiting People Demonstration Special Project (26-1-5-smjk-13-nsh).

Ethics Statement

This study only focuses on the characterization of carbon black filled natural rubber composites. No human participants, animal models, biological tissue samples or clinical experiments are involved in the whole research. Thus, ethical approval from research ethics committee is not required for this work.

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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