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I–V characteristics and electro-mechanical response of different carbon black/epoxy composites

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ABSTRACT

I–V characteristics and electro-mechanical response of carbon black (CB)/epoxy composites were studied experimentally. Two types of CB were used in the experiment, they were: sprayed CB and conductive CB particles. During the experiment, it was found that the *I–V* characteristics of the composites and their electro-mechanical response were greatly affected by the particle size of CB as well as their dispersion properties. The epoxy containing sprayed CB with the diameter of 123 nm composites gave predicted relationships, in terms of *I–V* characteristics and strain/electrical resistivity once they were subjected to a compressive load. The electrical breakdown assumption incorporated in the DC circuit model is proposed in this paper to interpret the response of the composites with different types of CB particles.

1. Introduction

Regardless of many existing successful applications of polymer composites, the fundamental and applied studies of such materials are still of acute interest [1–4]. A good example is the electrical properties of carbon black (CB) particles and CB filled polymer composites, which have been extensively studied in the past decades and is still interesting to many researchers. The electric properties of carbon powders under compression were experimentally studied [5,6]. The resistivity behaviors of CB-filled rubbers under monotonic static loading and cyclic loading were experimentally studied by Kost et al. [7–9]. The effects of pre-extension on electrical conductivity and mechanical properties of CB-filled rubber were studied by Hawhem et al. [10]. The electrical behavior of a CB-filled ethylene–octene elastomer under strain up to 500% was studied by Flandin et al. and a reversible and strain-rate independent decrease in resistivity up to 30% strain was found [11]. In 1964, non-ohmic *I–V* behaviors of CB-loaded rubbers were observed by Van Beek and Van Pul and the phenomenon was explained by internal field emission theory [12,13]. Non-ohmic *I–V* behavior of CB-loaded polyvinyl chloride was observed by Sichel et al. [14] in 1978 and the phenomenon was explained by thermal breakdown theory. On the other hand, the ohmic *I–V* behavior of CB-loaded rubber was observed by Ali and Abo-Hashem [15,16]. On the whole, the above researches mainly focused on the influence of the content and dispersion of CB on the electrical and electromechanical properties of the composites containing CB, but the influence of the diameter and structure of CB was insufficiently studied.

In this paper, an experimental investigation on the *I–V* characteristics and the electromechanical behaviors, i.e., electrical resistance-mechanical strain relations, of the epoxy matrix composites containing different types of CB are presented herein. The influence of the exposure to an electrical field, which may cause the electrical breakdown of epoxy resin, on the composites containing different types of CB will also be experimentally studied. A DC circuit model, which accommodates the possibility of electrical breakdown of the epoxy, will be proposed in the next section to facilitate the comprehension of experimental results.

2. Model for electric breakdown of conductive composites

The composite, which consists of two kinds of materials with different conductivities, can be simplified and represented by a DC circuit along the electric field direction when it is exposed to an electric field. The DC circuit consists of resistors with different resistances, which are connected in both series and parallel [17]. On the basis of the above knowledge, a DC circuit containing conductive chain unit in both series and parallel (see Fig. 1), is employed to interpret the microstructure of the conductive composites. A conductive chain unit consists of a CB chain and an epoxy resin gap, which are connected in series. When the com-

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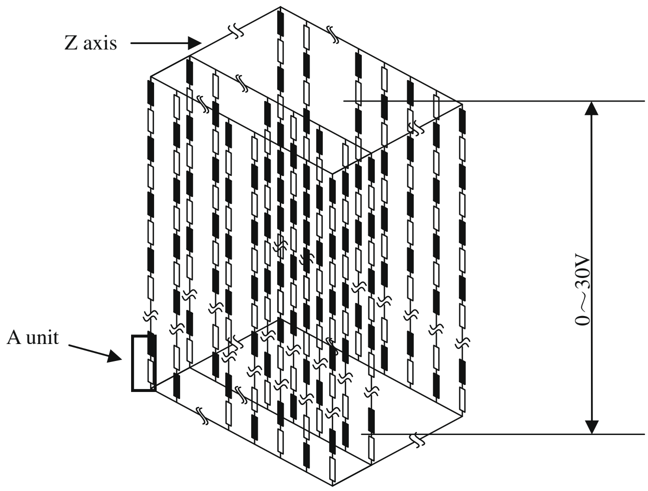

Fig. 1. The simplified modeling of structure of the composites exposed to an electric field.

posite is subjected to an electric field, the distribution of electric field in each conductive chain unit can be obtained [18].

$$E_1 = \frac{\sigma_2(d_1 + d_2)}{\sigma_1 d_2 + \sigma_2 d_1}E \qquad E_2 = \frac{\sigma_1(d_1 + d_2)}{\sigma_1 d_2 + \sigma_2 d_1}E \tag{1}$$

where E is the average field intensity applied on the conductive chain unit. E_1, σ_1, d_1 are the field intensity, conductivity, and thickness of a CB chain, respectively. Similarly, E_2, σ_2, d_2 are the field intensity, conductivity, and thickness of an epoxy resin gap, respectively.

CB particles usually aggregate into the chain formation in the composites. The electric field intensity at the apex of a CB agglomeration chain is much higher than at a single particle. The amplification factor β can be calculated by [19]

$$\beta = 3 + B \times \left(\frac{h}{d}\right)^D \tag{2}$$

where h is the length of a CB chain and d is the diameter of CB particle at the apex of a CB agglomeration chain, B and D are constants with the values $B = 2.947$ and $D = 0.7922$, respectively.

When the internal electric field of the composites reaches to a balance state under an external electric field, the current densities of the CB chains and the epoxy resin gaps are equal to each other at the interface

$$J_1 = \sigma_1 E_1 = J_2 = \sigma_2 E_2 \tag{3}$$

where J_1 and J_2 denote current densities of the CB chains and the epoxy resin gaps at the interface, respectively.

Consider that the electric field intensity of CB chain apex is enhanced by β times. Correspondingly, the electric field intensity of epoxy resin gap is also increased by β times. The conductivity of sprayed CB powders under compressive stress of 10 MPa is 2.86 Ω^{-1} cm^{-1} (provided by the company). The conductivity of conductive CB is 4.54 Ω^{-1} cm^{-1}, which is higher than that of sprayed CB. The conductivity of epoxy resins is 7.14×10^{-16} Ω^{-1} cm^{-1}. The ratio of conductivity between the CB chain and the epoxy resin gap is on the order of 4.0×10^{15} ($\sigma_1/\sigma_2 = 2.86 \times 10^{16}/7.14$) at least. Substituting this conductivity ratio into Eq. (1) and neglecting the smaller value $\sigma_2 \times d_1$, the amplified local field intensity on an epoxy resin gap can be approximated as

$$E_2 = \beta\frac{d_1 + d_2}{d_2}E \tag{4}$$

Assuming that the amplified local electric field intensity of the epoxy resin gap near the CB chain apex equals to the electrical breakdown limit of epoxy resin E_b, the maximum resin gap thickness (or the threshold resin gap thickness) that can be broken down by an electrical field E as:

$$d_{2,\text{threshold}} = \frac{\beta E d_1}{E_b - \beta E} \tag{5}$$

Eq. (5) suggests that whether a epoxy resin gap is broken down will be determined by the thickness of the epoxy resin gap, the diameter of CB particle, the length and shape of CB agglomeration, as well as the average electrical field applied.

3. Experiment

3.1. Materials and specimens

The conductive CB particles, which are hollow spheres with 33 nm in diameter and 1.5 nm thick wall, were obtained from Shandong Linzi Huaguang Chemical Industry Plant, China. The sprayed CB particles, which are solid spheres with diameter of 123 nm, were obtained from LiaoNin FuShun Chemical Industry Plant, China. Some properties of the two kinds of CB are shown in Table 1. Epoxy resin used in the test was E-51, made by Shenzhen Kunzhan Industrial Ltd., China. HK-021 liquid acid anhydride purchased from Wenzhou Qingming Chemical Ltd., China, was used as curing agent. *N,N*-Dimethylbenzylamine was used as accelerating agent, provided by Shanghai SSS Reagent Co. Ltd., China.

To fabricate the epoxy/CB composites, the epoxy resin and the curing agent were mixed in a vessel and then heated in an oven at 90 °C for 30 min. The CB particles and the accelerating agent were sequentially added into the mixture, which was stirred for 10 and 3 min, respectively. Then the mixture was poured into waxed molds in which four copper net electrodes had been inserted to form cubes of size 50 × 50 × 50 mm. The specimens were cured for 3 h at 90 °C, then 2 h at 120 °C and another 3 h at 150 °C. Finally, the specimens were cooled to room temperature in the oven before they were demolded. The compositions of the composite specimens are shown in Table 2. For each type of CB filler, there were three different CB loadings. Four specimens were prepared for each composition.

3.2. Test procedure

The electromechanical behavior was tested under a hydraulic mechanical testing system (MTS) with 120 kN maximum load capacity. During the compressive tests, two strain gauges attached on the samples surface was used to test the strain of samples and

Table 1
Properties of carbon black.

Parameter	Diameter (nm)	Iodine absorption value (g/kg)	DBP value (cm^3/100 g)	Specific area of BET (m^2/g)	Density (g/cm^3)	Electrical conductivity (Ω^{-1} cm^{-1})
Conductive CB (hollow sphere)	33	1070	380	1056	0.26	4.54
Sprayed CB (solid sphere)	123	15	120	32	1.8	2.86

Table 2
Composition of composites.

Composition No.	C1	C2	C3	S1	S2	S3
Epoxy resin	100	100	100	100	100	100
Curing agent	75	75	75	75	75	75
Accelerating agent	1	1	1	1	1	1
Conductive CB (by weight)	5.25	3.5	1.75			
Conductive CB (by volume)	24.63	16.42	8.21			
Sprayed CB (by weight)				52.5	43.75	35
Sprayed CB (by volume)				35.58	29.64	23.73

the resistance of samples was tested by four-probe method. A data acquisition module ADAM-4018 and a DH1718D-4DUAL power supplier were used to measure the resistance. To avoid the unplanned permanent microstructural change during the first round tests, the voltage applied by the power supplier was set to be less than 0.5 V to characterize the electrical resistance-mechanical compressive strain relation.

To investigate the *I–V* behaviors of the composites, the specimens were exposed to an electric field with various voltage levels. The power was given by the DH1718D-4DUAL power supplier. The current intensity was measured at each voltage level by using a DHC3P digital ampere meter fabricated by WenZhou DaHua Instruments Co. Ltd. For every composition, four original specimens were available for testing. Firstly, all original specimens of each composition were tested under MTS and the electromechanical behaviors of the composites were recorded. Secondly, four specimens of each composition were exposed to an electric field with the voltage gradually increased from 0 to 30 V and their *I–V* behaviors were recorded.

4. Results and discussion

4.1. Electrical properties of the original composites

The resistivity of composites containing different CB changes with the volume fraction of CB is showed in Fig. 2.

Comparing Fig. 2(a) and (b) we can see that the resistivity of composites containing conductive CB is less than that of composites containing sprayed CB while the content of CB is same. It can be seen from Fig. 2 that for the two composites containing conductive CB and containing sprayed CB, respectively, their relationship between the resistivity and the volume fraction of CB is totally different. The resistivity of composites containing conductive CB decreases gradually with the increasing of content of CB, and that there is no percolation property can be found. Moreover, the resistivity decreases a lot with the little change of content of CB, when the content of conductive CB is low. However, the relationship between the resistivity of composites and the volume fraction of CB for the composites containing sprayed CB exhibits percolating properties. First, the resistivity decreases slowly with the increasing of content of CB, then the resistivity decreases sharply when the content of CB reached percolation threshold. Again, when the content of CB is higher than percolation threshold, the resistivity of composites containing sprayed CB decreased slowly with the increasing of content of CB. In this paper, the percolation threshold of composites containing sprayed CB is 15% volume fraction, this value is the same as that of the classical statistical percolation model established by Zallen et al. The different electrical properties of composites containing conductive CB and sprayed CB will be interpreted in Section 4.4.

4.2. Electromechanical behaviors of the original composites

The relations of the change in electric resistance and applied compressive strain of the original composites containing conductive CB are presented in Fig. 3 (denoted by $\Delta R/R_0$, ΔR is the variation of electric resistance and R_0 is the initial electric resistance). It can be seen from Fig. 3 that the results depend on the composition of the composite. The resistance fluctuates as the compressive strain increases. The relations of the change in electric resistance and applied compressive strain of the original composites containing sprayed CB are presented in Fig. 4. It can be seen from Fig. 4 that the resistances of all the composites decrease monotonously when the compressive strain increases and the decreased value of resistance depend on the concentration of CB particles. Comparing Fig. 3 with Fig. 4, significant differences in the electromechanical behaviors between the composites containing conductive CB and the composites containing sprayed CB can be observed. This phenomenon will be interpreted in Section 4.4.

4.3. I–V characteristics of composites (electric field exposure)

Four specimens of each composition unloaded from the MTS were exposed to an electric field with the voltage applied gradually increased from 0 to 30 V. The distance between the positive and negative electrode was 2 cm, so the electric field intensity varied from 0 to 15 V/cm. The current was measured by the DHC3P digital ampere meter at each voltage level. The voltage–current relations of the specimens containing conductive CB and the ones containing sprayed CB are shown in Figs. 5 and 6, respectively. We find that

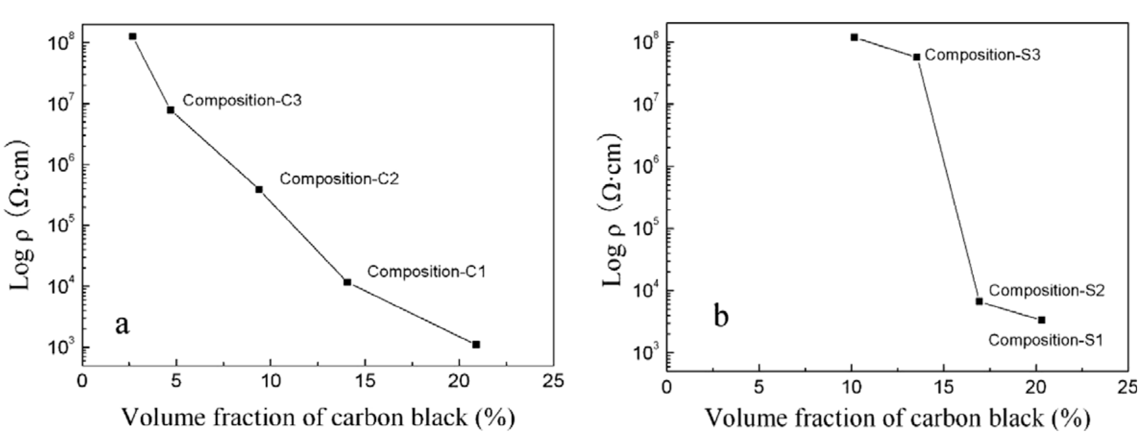

Fig. 2. The concentration dependence of the resistivity (a) composites containing conductive CB (b) composites containing sprayed CB.

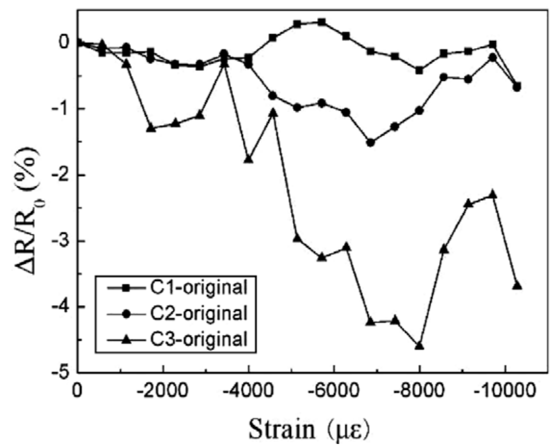

Fig. 3. The fractional change in resistance versus the compressive strain of the original composite specimens filled with conductive CB.

the *I–V* characteristics of the composites containing conductive CB is nonlinear (see Fig. 5), which significantly differ from the linear *I–V* characteristics of the composites containing sprayed CB (see Fig. 6).

The resistivities of the specimens were measured before and after the electric field exposure. The resistivities of the conductive CB specimens pre- and post-exposed to the electric field (15 V/cm), denoted by ρ_{pr} and ρ_{po}, respectively, are listed in Table 3. It can be seen from Table 3 that the resistivities of the specimens containing conductive CB dramatically decrease after being exposed to the electric field. The ratios of resistivity decrease vary with CB content, e.g. 1/60.39, 1/28.02 and 1/109.21 times for compositions C1, C2 and C3, respectively. The decrease of the resistivity of the composites is irrecoverable and this phenomenon has not been reported by any other researchers before. Note that there are no change in the resistivities of the specimens contained sprayed CB before and after the electric field exposure.

According to the model in Section 2, it can be concluded that the composites containing the conductive CB (small particle diameter, long catenulate CB agglomerations and thin epoxy resin gaps) can be broken down by an electrical field more easily. The conductive CBs with a small particle diameter and high structure can easily form long catenulate agglomerations in the matrix [12]. On the other hand, the sprayed CB have a bigger particle diameter and low structure and usually form global agglomerations in the matrix [12]. These can be proved by the TEM images of the conductive CB

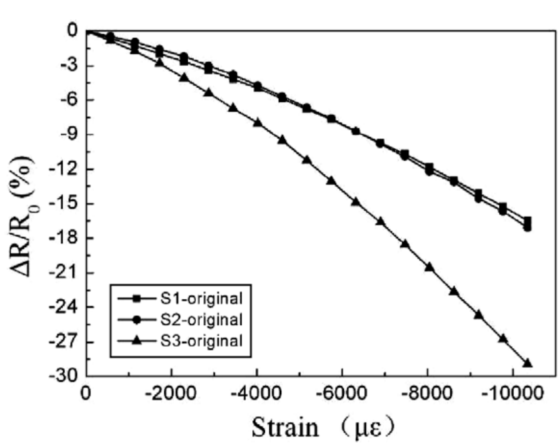

Fig. 4. The fractional change in resistance versus the compressive strain of the original composite specimens filled with sprayed CB.

and sprayed CB agglomerations which are showed in Fig. 7. According to Fig. 7, it can be assumed that the diameter of a conductive CB agglomeration chain at the apex is the same as that of a single particle (i.e., $d = 33$ nm). According to Ref. [14], the even length of CB agglomeration chain (d_1) is 1.0 μm and the length of conductive CB agglomeration showed in Fig. 7 is 1.3 μm, so it can be assumed that the length of conductive CB agglomeration is 1.0 μm. The electrical breakdown limit of epoxy resin (E_b) is 200 kV/cm (provided by the company). Substituting these values into Eq. (5), it can be obtained that the epoxy resin gap in a conductive CB composite can be broken down by the 15 V/cm electric field if its thickness is less than 3.5 nm (i.e., the threshold resin gap thickness). According to Fig. 7, it also can assumed that the diameter of a sprayed CB agglomeration chain at the apex is the same as that of a single particle (i.e., $d = 123$ nm) and the length of sprayed CB agglomeration is 2 μm. With similar calculation, it can be obtained that the breakdown threshold resin gap thickness for the composites containing sprayed CB under the same 15 V/cm electrical field is 4.48 nm. The breakdown threshold thickness of an epoxy resin gap increases as the length of a CB agglomeration chain increases and as the diameter of a CB particle decreases. On the other hand, the thickness of epoxy resin gap between CB particles can be calculated by the following equation [20]:

$$S = \sqrt[3]{\frac{1}{6}\pi d_A^3 + \frac{\frac{1}{6}\pi d_A^3 \rho_c 100}{L\rho_p}} - d_A \tag{6}$$

where S is distance of CB particle separation; L is the number of parts of CB by per hundred of polymer; ρ_c and ρ_p are the densities of CB particle and polymer, respectively; $d_A = 6/(\rho_c \cdot A)$, A is the Specific area of CB particle. Substituting the corresponding values into Eq. (6), it can be calculated that the distance of CB particle separation of the specimens containing conductive CB is 0.45–6.32 nm and that of the specimens containing sprayed CB is 64.9–86.42 nm.

Eq. (6) is based on the assumption that the carbon particles are arranged as in a cubic lattice. However, the CB particles are actually agglomerated in composites as evidenced by the SEM photographs shown in Fig. 8. It can be observed that the thickness of epoxy resin gaps of composites containing sprayed CB is very large, reaching several hundred nanometers, and that of the specimens containing conductive CB is very small and the thickness of the epoxy resin gaps is too small to be recognized in the SEM picture shown in Fig. 8. Furthermore, Fig. 8(c) shows that the CBs distribute in the sprayed CB composite specimen uniformly. On the other hand, as shown in Fig. 8(d), the conductive CB composite specimen consists of CB and resin mixture and pure resin regions. As a result, the thickness of epoxy resin gaps between two conductive CB agglomeration chains is less than that of the composites in which the CB is dispersed uniformly. Consequently, the thickness of the epoxy resin gaps between two CB agglomeration chains of composites containing conductive CB should be less than the thickness of the gaps calculated from Eq. (6).

When the composites containing conductive CB being exposed to an electric field, part of epoxy resin gaps whose thickness are less than 3.5 nm will be broken down gradually. This epoxy gap breakdown process will lead to the non-linear *I–V* characteristics of the composites. When the composites containing sprayed CB being exposed to the same electric field, no epoxy resin gaps will be broken down and the *I–V* characteristics of composites will be linear because that all the epoxy resin gaps of the composites are larger than 4.48 nm. An epoxy resin gap that is broken down converts from an insulator into a conductor. As a result, as seen in Table 3, the resistivity of a conductive CB composite irrecoverably and dramatically drops after the electrical field exposure.

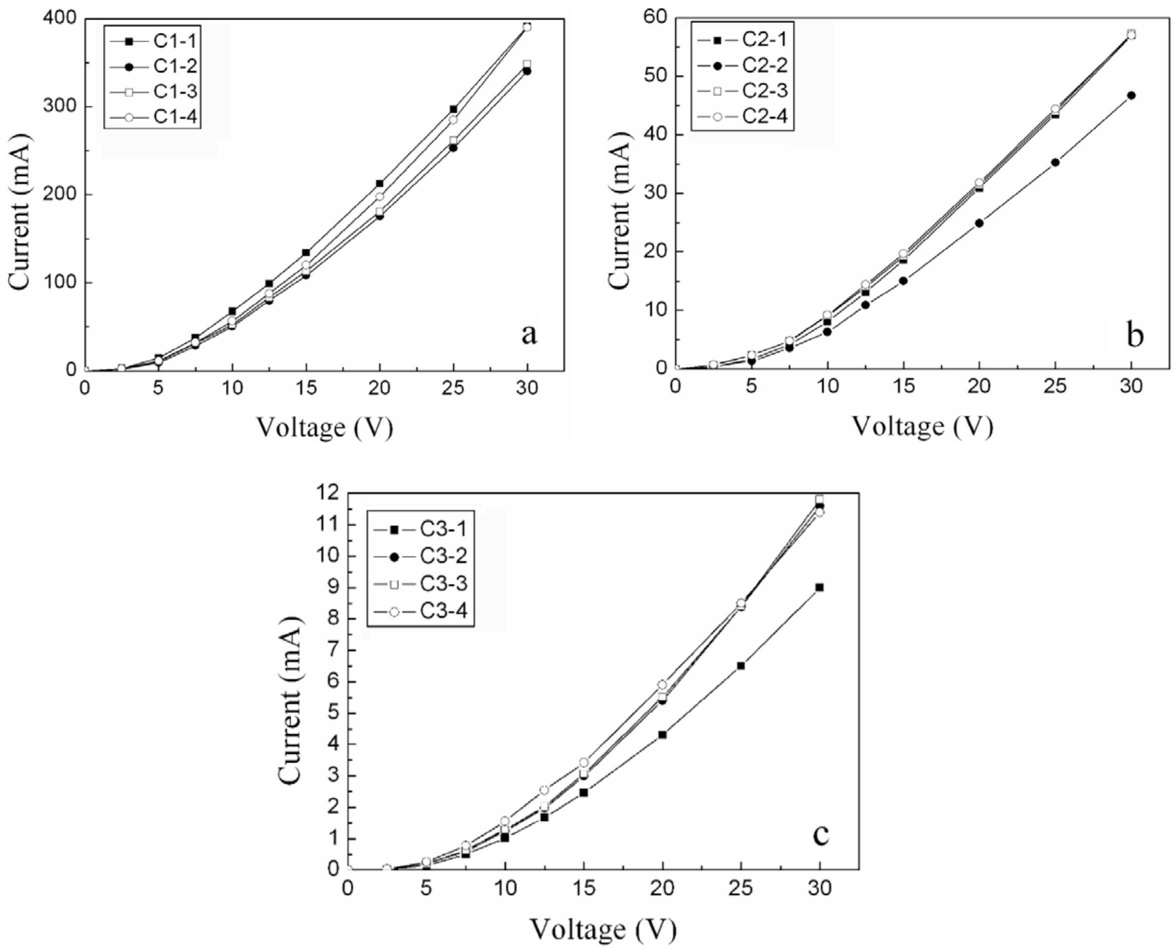

Fig. 5. Voltage–current relations of specimens containing conductive CB. (a) The composites containing 24.63% CB (by volume). (b) The composites containing 16.42% CB (by volume). (c) The composites containing 8.21% CB (by volume).

4.4. Interpretation of the electrical and electromechanical properties of composites pre-exposed to an electric field

With high structure and huge specific area, conductive CB can easily form long catenulate agglomerations in the matrix. Even the content of CB is very low, the conductive CB still can form long conductive chains at few areas of the composites. At the other area of composites, there are no CB can be found, just like the image showed in Fig. 8(d). So, even the volume fraction of CB is only 4.69% the conductivity of composites containing conductive CB is 15 times as that of composites containing 10.16% volume fraction of sprayed CB. With the increase of CB content, the number of conductive chains formed by conductive CB increase. These conductive chains connect each other by thin epoxy resin gaps and form conductive channel throughout the whole composites. Although the resistivity of these conductive channels is very small, the macroscopic resistance of composites does not decrease sharply like percolation because the conductive channels only exist at few areas of the composites and at other areas of the composites even no carbon black can be found. With the content of carbon black increase further the number of conductive channel increase and the resistance of composites decrease further more.

With the low structure and small specific area sprayed CB are easily form global agglomerations and disperse in the matrix uniformly (Fig. 8(c)). When the content of CB is less than the percolation threshold of composites, the resistance of composites is affected by two factors: (1) With the increase of CB content the CB is more easily to aggregate. Due to the agglomerations of sprayed CB are global the aggregate of CB will increase the thickness of epoxy resin gaps between CB agglomerations and this will increase the resistance of composites. (2) The increase of CB content will increase the number of CB particles. The increase of CB particles' number will decrease the thickness of epoxy resin gaps between CB agglomerations and this will decrease the resistance of composites. Therefore, affected by these two factors, the resistance of composites decreases slowly with the increase of content of CB when the content of CB is less than the percolation threshold. When the content of CB reaches the percolation threshold, the distance between two sprayed CB agglomerations decrease with the increasing of content of CB. Then the conductive channels are formed and this lead to the sharp decreases of the composites' resistance. When the content of CB is more than percolation threshold, the thickness of epoxy resin gaps between CB agglomerations will not decrease further more [21]. So, the resistance of composite increase slowly with the increasing of CB content.

When a composite is subjected to the compression, the compressive deformation along the compression direction coexists with the Poisson's ratio induced expansion, which is perpendicular to the compression direction. Therefore, the microstructure of the composite should deform and move correspondingly. If the CB agglomerations are global in a composite, such as the sprayed CB composite, the compression will move the CB agglomerations close to each other as shown in Fig. 9(a) and (b). As a result, the resistance of the sprayed CB composite will be reduced under the

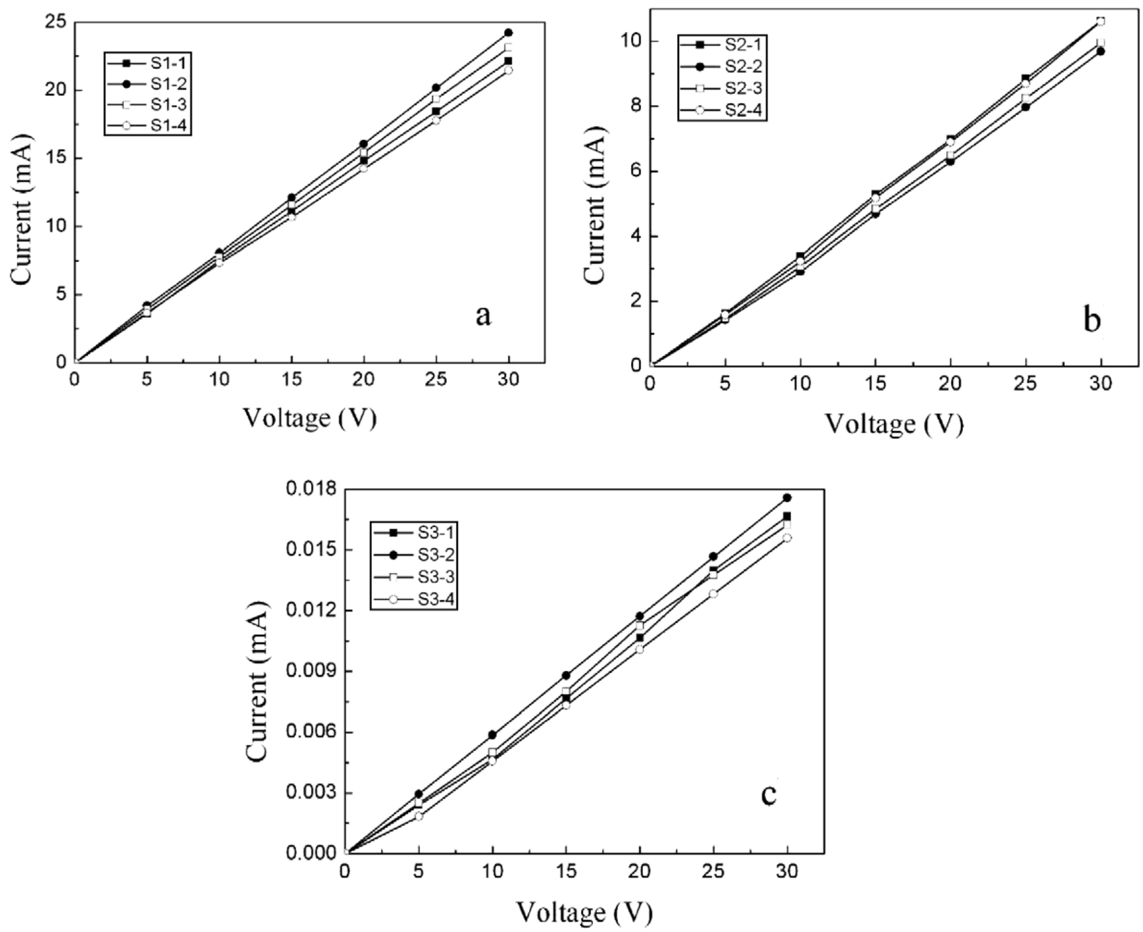

Fig. 6. Voltage–current relations of specimens containing sprayed CB. (a) The composites containing 35.58% CB (by volume). (b) The composites containing 29.64% CB (by volume). (c) The composites containing 23.73% CB (by volume).

Table 3
Electric resistivities of the specimens containing conductive CB pre- and post-exposed to the electric field.

Composition No.	C1	C2	C3	S1	S2	S3
ρ_{pr} (Ω cm)	11,575	39,050	777,250	3325	16,653	5,595,000
ρ_{po} (Ω cm)	191.5	1385	6932.5	3325	16,653	5,595,000
ρ_{po}/ρ_{pr}	1/60.39	1/28.02	1/109.21	1	1	1

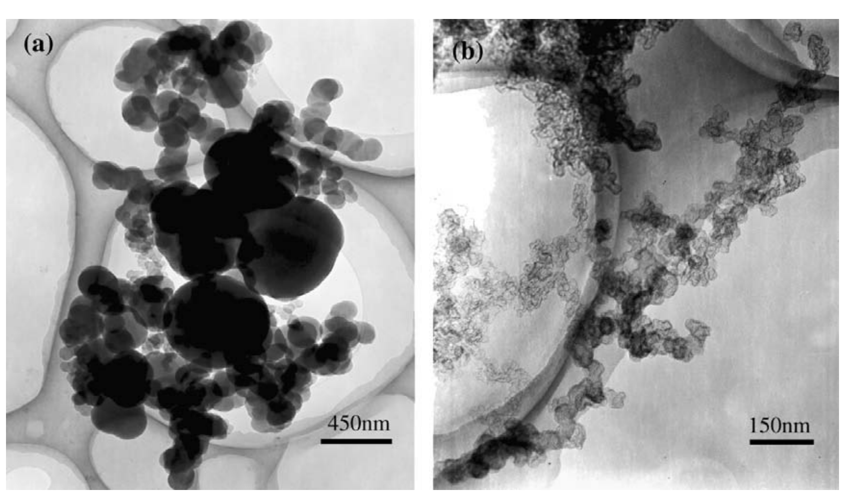

Fig. 7. The TEM images of CB agglomerations. (a) Sprayed CB. (b) Conductive CB.

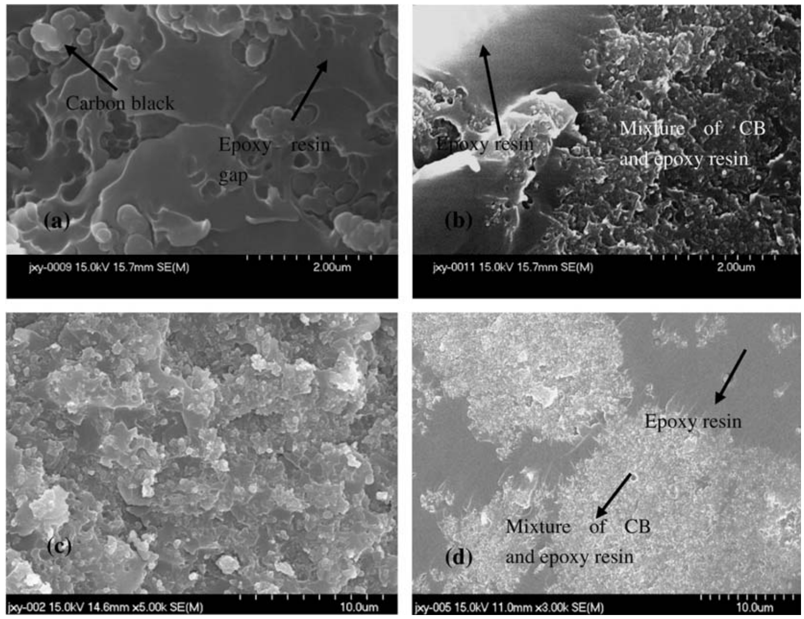

Fig. 8. The SEM pictures of composite specimens. (a) and (c) Composites containing sprayed CB. (b) and (d) Composites containing conductive CB.

compression as shown in Fig. 4. On the other hand, if the CB agglomerations are catenulate in a composite, such as the conductive CB composite, the compression will simultaneously move and rotate the CB agglomerations (see Fig. 9(c) and (d)). The rotation of catenulate CB agglomerations which is caused by the deformation perpendicular to the compression direction will fluctuate the resistance of the conductive CB composites as shown in Fig. 3. Based on analysis mentioned above, it is concluded that the electromechanical properties of composites are affected by the diameter and structure of the CB particles. Hence, the composites can be tailored for the desirable electromechanical performance by selecting the diameter and structure of the CB particles.

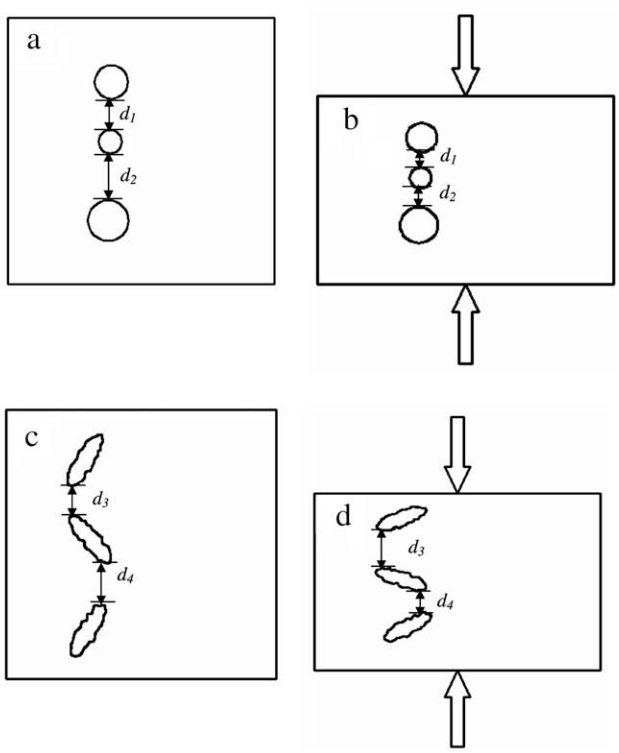

Fig. 9. The compression models of the composites containing different types of CB. (a) and (b) The composites containing sprayed CB. (c) and (d) The composites containing conductive CB.

5. Conclusion

In this paper, an experimental study for the electrical and electromechanical behaviors of two different CB filled epoxy matrix composites is presented. A DC circuit model capable of incorporating local epoxy electrical breakdown is proposed to explain the results.

According to this model, whether an epoxy resin gap between two CB agglomerations in the composites is broken down should be determined by the thickness of the epoxy resin gap, the diameter of CB particles and the lengths and shapes of CB agglomerations. The epoxy resin gaps of a sprayed CB composite cannot be broken down easily by the electric field since this kind of composites have thicker epoxy resin gaps, larger CB diameter, and are easy to form global CB agglomerations. Therefore, the *I–V* characteristic of a composite containing sprayed CB is linear and there is no difference between the specimens with or without electric field treatment. On the other hand, a composite containing conductive CB has thinner epoxy resin gaps, smaller CB diameter, and is easy to form catenulate CB agglomerations. If this conductive CB composite is under an electric field, some thin epoxy resin gaps can be easily broken down. Since the epoxy resin gaps that are broken down will change into conductors, the resistivity of the conductive CB composite will be irrecoverably and dramatically reduced by the electric field treatment.

Relationships between the resistivity of CB filled epoxy matrix composites and their CB content are different for the composites with various CB. The resistivity of composites containing conductive CB decreases gradually with the increasing of content of CB, and there is no percolation property can be found. However, the relationship between the resistivity of composites and the volume fraction of CB for the composites containing sprayed CB exhibits percolation property. It is explained that the different way forming conductive channels by CB in the composites containing different CB cause the different electrical behaviors.

In this paper, interesting results are also found in the electromechanical behaviors of the CB filled epoxy matrix composites. As being gradually loaded with compressive strain, the electrical resistance of a sprayed CB filled epoxy composite will monotonously decrease accordingly. However, this kind of decrease is not found in the conductive CB composites. It is hypothesized that the rotation of the catenulate CB agglomerations in the conductive CB composites causes the non-monotonic change in the distance between two catenulate CB agglomerations thus makes the fluctuation in the electrical resistance of the conductive CB composites.

Acknowledgments

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