



# Effect of nano-fillers on nonlinear conduction and DC breakdown characteristics of epoxy composites

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## Abstract

In order to improve the nonlinear conduction and dc breakdown strength, the influences of nano-fillers on epoxy nano-composites are studied. The epoxy nano-composites are prepared by regulating the shape factor of the semiconductor filling phase, with separate doping or two phase co-doping. The crystal structure and morphology of the composites and nano-fillers are analyzed by X ray diffraction and scanning electron microscopy. The conductivity and dc breakdown strength of the nano-composites are measured. The results show that the surface treated nano-fillers are well dispersed in the epoxy matrix. Compared with pure epoxy and epoxy nano-composites with lower filler loading of ZnO particles, the ones with higher filler loading of ZnO particles and lower filler loading of ZnO sheets exhibit a distinct nonlinear conduction character, but their breakdown strength is significantly reduced. By contrast, the breakdown strength of BN/epoxy nano-composites with low filler loading is significantly increased, and the 3 wt% BN/epoxy nano-composite behaves the highest breakdown strength. Based on 3 wt% BN nano-sheet, ZnO–BN epoxy nano-composites are prepared by two phase co-doping. Compared with single doping, the two phase co-doping nano-composites can not only improve the nonlinear conductivity, but also alleviate the problem of the serious deterioration of dc breakdown performance. The nonlinear conduction and dc breakdown properties of the studied nano-composites are related to the interaction zone of nano-filler in polymer matrix.

## 1 Introduction

Epoxy resin (EP) as an important polymer insulation has been widely used in electrical and electronic engineering due to its excellent mechanical and electrical properties [1, 2]. For instance, EP can be used as main insulation and supporting insulation of electrical equipments [3]. However, space charges are usually formed in EP and its composites under high electric field, especially high dc electric field. The space charge accumulation will lead to the ageing or insulation failure of polymer composites [4, 5].

It is reported that the application of composites with nonlinear conductivity is an effective way to deal with the space charge accumulation in polymer materials [6, 7]. The intrinsic conduction of pure polymer materials usually behaves a weak nonlinear characteristic. In order to improve the

nonlinear conductivity of polymer materials, the inorganic conductor or semiconductor powders are usually applied as fillers [8, 9]. The conductivity of polycarbonatediol polyurethanes remains at the level of  $10^{-14}$  S/cm when the content of conductive expanded graphite (EG) is in the range of 10 to 20 wt%, while an abrupt increase in conductivity is observed when the EG content is close to 30 wt% [8]. Comparing with pure silicon rubber (SR), the 45 wt% SiC/SR nano-composite exhibits a very strong nonlinear conduction characteristic, and it can effectively homogenize the electric field distribution of cable termination [9]. In recent decades, the researches on the nonlinear conduction characteristics of polymer composites are mainly focused on the single doping strategy. By doping a single kind of nano-filler such as ZnO or SiC into the polymer matrix, the nonlinear conductivity of the nano-composites can be improved [10–12]. In general, when the filler loading is very high, the composite would show a stronger nonlinear conduction characteristic, which is very helpful to suppress the space charge formation and to alleviate the effect of electric field concentration [13–15]. However, the breakdown strength degradation of such composites is obvious because of the high filler loading. The strong nonlinear conduction of the composites is

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always obtained at the expense of breakdown strength. Most of the researches are merely focused on the nonlinear conductivity characteristics and space charge suppression effect of the nonlinear conduction composites [6, 7, 10–12], but the breakdown strength of the nonlinear conduction composites are rarely reported [16]. Therefore, it is necessary to seek a strategy for solving the paradox between the excellent nonlinear conductivity and low breakdown strength of the nonlinear conduction composites.

The purpose of this work is to elucidate the influence of ZnO nano-particles (ZnO NPs), ZnO nano-sheets (ZnO NSs) and boron nitride nano-sheets (BN NSs) on the epoxy nano-composites. The nonlinear conduction and breakdown strength of such nano-composites are discussed in details, respectively. The results show that the single doping of ZnO NPs or ZnO NSs can improve the nonlinear conductivity of epoxy nano-composites. Considering that the nonlinear conduction characteristics of the ZnO NPs/EP or ZnO NSs/EP nano-composites are obtained at the expense of breakdown strength, the two phase co-doping strategy are used to improve the nonlinear conduction and breakdown strength of the epoxy nano-composite by the co-doping of BN NSs and ZnO NPs or ZnO NSs.

## 2 Experiment sections

### 2.1 Materials

The E-51 epoxy resin is used as the polymer matrix and the Methyl Tetra Hydro Phthalic Anhydride (MTHPA) is used as the hardener. The weight ratio of the matrix and the hardener is 100:85. The commercially available ZnO NPs, ZnO NSs and BN NSs are used as the fillers, respectively. All of the nano-fillers are surface modified by silane coupling agent. The filler loadings of ZnO NPs are 30 and 40 wt%, respectively, and that of ZnO NSs is 30 wt%. While the filler loadings of BN NSs are 1, 3, 5 and 7 wt%. The filling loadings of the two phase co-doping epoxy composites are 30 wt% ZnO NPs/3 wt% BN, 40 wt% ZnO NPs/3 wt% BN and 30 wt% ZnO NSs/3 wt% BN, respectively.

### 2.2 Preparation of epoxy nano-composites

The surface modified nanofillers (such as ZnO NPs or ZnO NSs) are added into epoxy resin matrix and are mixed by high-speed shearing for 20 min. The hardener is then added in the mixture of nanofillers and EP matrix. The electrical blending and ultrasonic mixing are applied for 30 min. After degassing, casting and curing, the epoxy nano-composites are then prepared. The curing process is achieved by three-step curing temperature, namely, 80 °C for 2 h, 100 °C for 2 h and 120 °C for 6 h. A large number of specimens are

made. Among them, 20 seemingly perfect specimens for each filler loading are selected for testing. The average thickness of the specimens is about 0.3 mm.

### 2.3 Characterization method

X-ray diffraction (XRD) is used to detect the phase composition of epoxy nano-composites under 40 kV and 40 mA. Field emission scanning electron microscopic (FESEM) is applied to observe the morphology of the fillers and epoxy nano-composites, and the acceleration voltage is 5 kV. The flat sections of specimens are treated by gold sputtering method for better observation.

The dc conductivities of specimens are measured by dc conductance measurement system, which is made up of a high voltage dc power supply, a picoammeter and a three-electrode system. The diameters of the measuring electrode and the gap distance between the measuring electrode and the ground electrode are 50 mm and 2 mm, respectively. The applied electric field is from 1 to 15 kV/mm. The dc current density is obtained when the electric field is applied on the specimen for 30 min. According to the geometry structure of specimen, the dc conductivity is then determined.

A sphere–sphere electrode system is applied to evaluate the dc breakdown strength of epoxy nano-composites. Both of the sphere electrodes are immersed in a kind of insulating oil to avoid surface flashover. The diameters of the sphere electrodes are 25 mm, respectively. The voltage rise rate is 1 kV/s. A set of 10 specimens for each filler loading are tested and used to calculate the breakdown strength. Two-parameter Weibull distribution is used to evaluate the breakdown behavior of the composites with different filler loading.

## 3 Results and discussion

### 3.1 XRD curves

The XRD analysis is used to identify the phase structure of the composites. Fig 1a shows the XRD patterns of inorganic fillers, including ZnO NPs, ZnO NSs and BN NSs after surface modification. The characteristic peaks of ZnO and BN can be indexed, which are consistent with [PDF 00-036-1451] and [PDF 00-045-0896], respectively. For example, the (100), (002), (101) and (102) diffraction peaks of ZnO NPs and ZnO NSs emerge at  $2\theta$  of 31.73°, 34.4°, 36.21° and 47.51°, respectively. Meanwhile, the (002) diffraction peak of BN NSs is observed at  $2\theta$  of 26.8°. The XRD patterns of ZnO NPs/EP, ZnO NSs/EP, and ZnO NSs-BN NSs/EP composite are shown in Fig. 1b. The characteristic diffraction peak at  $2\theta$  of 16° can be observed and attributed to pure EP [17]. Combined with the XRD patterns of inorganic fillers as

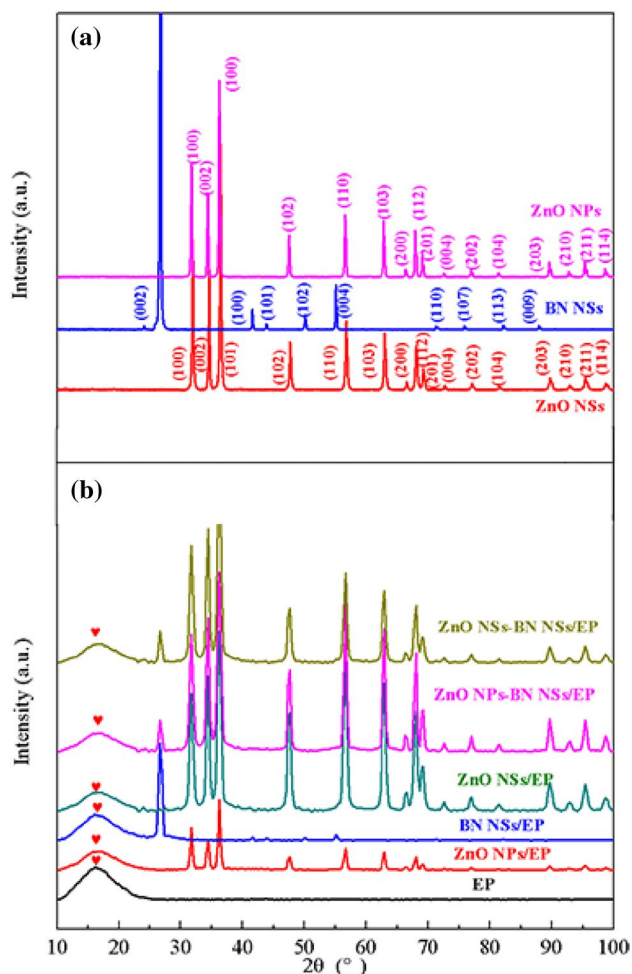


Fig. 1 XRD test data

shown in Fig. 1a, the diffraction peaks of both EP matrix and inorganic fillers can be clearly detected in the composites, which indicates that the fillers are physically mixed with EP and no impurity phase is introduced.

### 3.2 SEM image

Figure 2 shows the field emission scanning electron microscope images and size distributions of the three fillers. Figure 2a, c and e are ZnO NPs, ZnO NSs and BN NSs respectively, and Fig. 2b, d and f are the corresponding size distribution of them. ZnO NPs is granular and the average particle size is about 300 nm in Fig. 2a, b, and ZnO NSs presents a two-dimensional sheet structure with a diameter of about 150 nm in Fig. 2c, d. Figure 2d, e show two-dimensional sheet structure of BN NSs after ultrasonic peeling, with an average diameter of about 3–5  $\mu\text{m}$ .

Figure 3 shows the field emission scanning electron microscope images of the samples. Figure 3a is pure EP, Fig. 3b–d are 40 wt% ZnO NPs/EP, 30 wt% ZnO NSs/EP,

and 3 wt% BN NSs/EP samples respectively. Figure 3e, f are 40 wt% ZnO NPs-3 wt% BN NSs/EP and 30 wt% ZnO NSs-3 wt% BN NSs/EP samples.

SEM analysis can be used to obtain the microscopic morphology of composites effectively. An even and uniform section can be observed in the SEM image of pure epoxy as shown in Fig. 3a. ZnO NPs are well dispersed in the epoxy matrix, and only a few large agglomerates can be observed in Fig. 3b. By contrast, as shown in Fig. 3c, d, the fillers morphology is significantly different from that in Fig. 3b. They all presented two-dimensional sheet structure and are evenly distributed in epoxy matrix, where ZnO NSs are small and dense while BN NSs are large and sparse. In Fig. 3e, f, in addition to uniformly distributing ZnO NPs and ZnO NSs, a small amount of two-dimensional sheet structure BN NSs with larger size are observed obviously in the composite sample, which are randomly and uniformly embedded in the composite material, and there are also no obvious holes or defects on the surface of the composite material.

### 3.3 The conductivity and dc breakdown performance

The dc conductivity may be dependent on the applied electric field and behave nonlinear characteristic. In order to describe the nonlinear degree, the following equation is usually used [18].

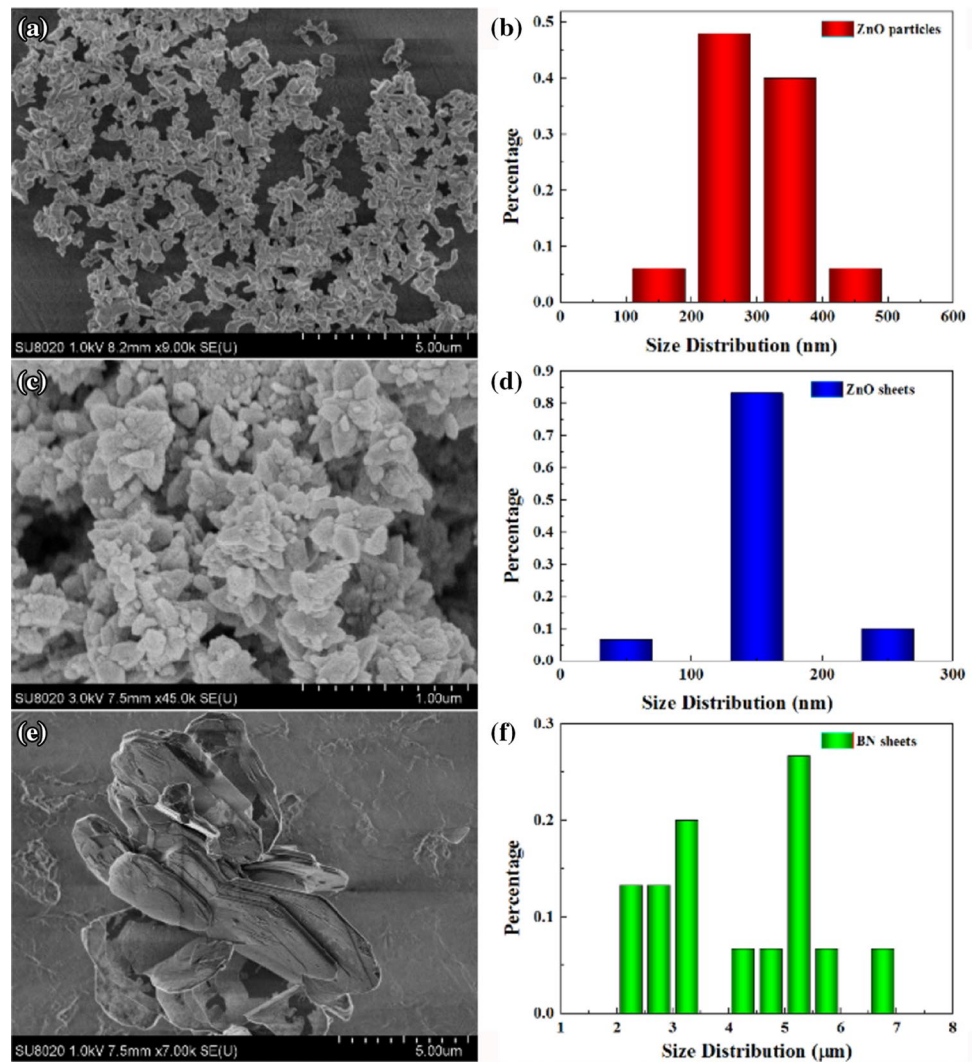
$$\gamma = \gamma_0 (E/E_0)^{\alpha_n} \quad (1)$$

where  $\gamma$  is conductivity.  $\gamma_0$  is the switching conductivity.  $E$  is the applied electric field.  $E_0$  is the switching electric field.  $\alpha_n$  is the nonlinearity exponent and it is used to define the nonlinear degree of the dc conductivity.

The relationship between the dc conductivity and the applied electric field of ZnO/EP nano-composites with different shape factors and different filler loading is shown in Fig. 4. For pure epoxy and 30 wt% ZnO NPs/EP, the dc conductivity behaves as a very weak nonlinear function of the applied electric field. Similarly, both of 40 wt% ZnO NPs/EP and 30 wt% ZnO NSs/EP also show a very weak dependence on the electric field when the maximum electric field is less than 5 kV/mm. In contrast, when the electric field is higher than 5 kV/mm, both of the last two nano-composites present a very strong nonlinear relationship between the dc conductivity against the applied electric field. The  $\alpha_n$  is 4.87 and 6.92, and the  $E_0$  is 6.12 kV/mm and 5.25 kV/mm, respectively. These results indicate that the adjustment of the shape factor of semiconductor filling phase can reduce the filler loading of the fillers and improve the nonlinear characteristics of the composites.

Base on the multi-region structure model [19], when the nano-particles are added into the epoxy resin, the interaction

**Fig. 2** SEM images and size distributions of the three fillers



zone is then formed between the matrix and the nano-particles. For ZnO NPs nano-composites, a lower filler loading of ZnO NPs may introduce plenty of deep trap sites into the interaction zone, and then the free carriers are captured by the deep trap sites and cannot escape during a long time. Thus the dependence of dc conductivity on the applied electric field is weakened due to the much lower density and mobility of free carriers. In contrast, the increasing filler loading may make the interaction zone overlap. The thickness of the interaction zone is equivalent to be enlarged. In the thicker interaction zone, the carriers can gain more energy to migrate further. Longer migration distance makes the average density and mobility of free carriers increase. So the nonlinear conductivity characteristics becomes more stronger. For ZnO NSs nano-composites, the unique two-dimensional sheet structure of ZnO NSs maybe introduce a large number of shallow trap sites into the interaction zone. Due to the limited binding capacity of shallow trap sites to carrier, when the electric field increases, the carrier is easier

to escape from the trap. Then the free carrier density and mobility are higher, so the nonlinearity exponent of the sample is larger at lower filler loading of ZnO NSs. This is also why the threshold field strength of the ZnO NSs composites shows a decreasing trend.

The dc breakdown strength of epoxy nano-composites are estimated according to two-parameter Weibull distribution equation defined as follows, and the curves are shown in Fig. 5.

$$P(E) = 1 - \exp \left[ -\left( \frac{E}{E_0} \right)^\beta \right] \quad (2)$$

where  $P(E)$  is the probability of cumulative failure.  $\beta$  is the shape parameter.

Usually, the logarithmic deformation as follows is taken from both sides of Eq. (2) to obtain the linear regression equation and to determine the Weibull distribution parameters.

$$\ln \{ -\ln [1 - P(E)] \} = \beta (\ln E - \ln E_0) \quad (3)$$

Fig. 3 SEM images of samples

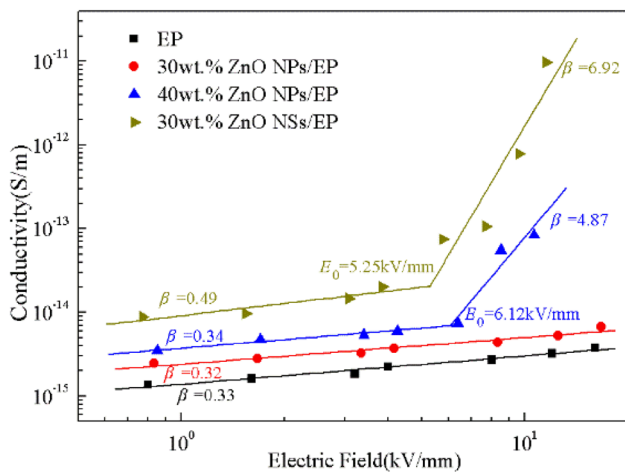
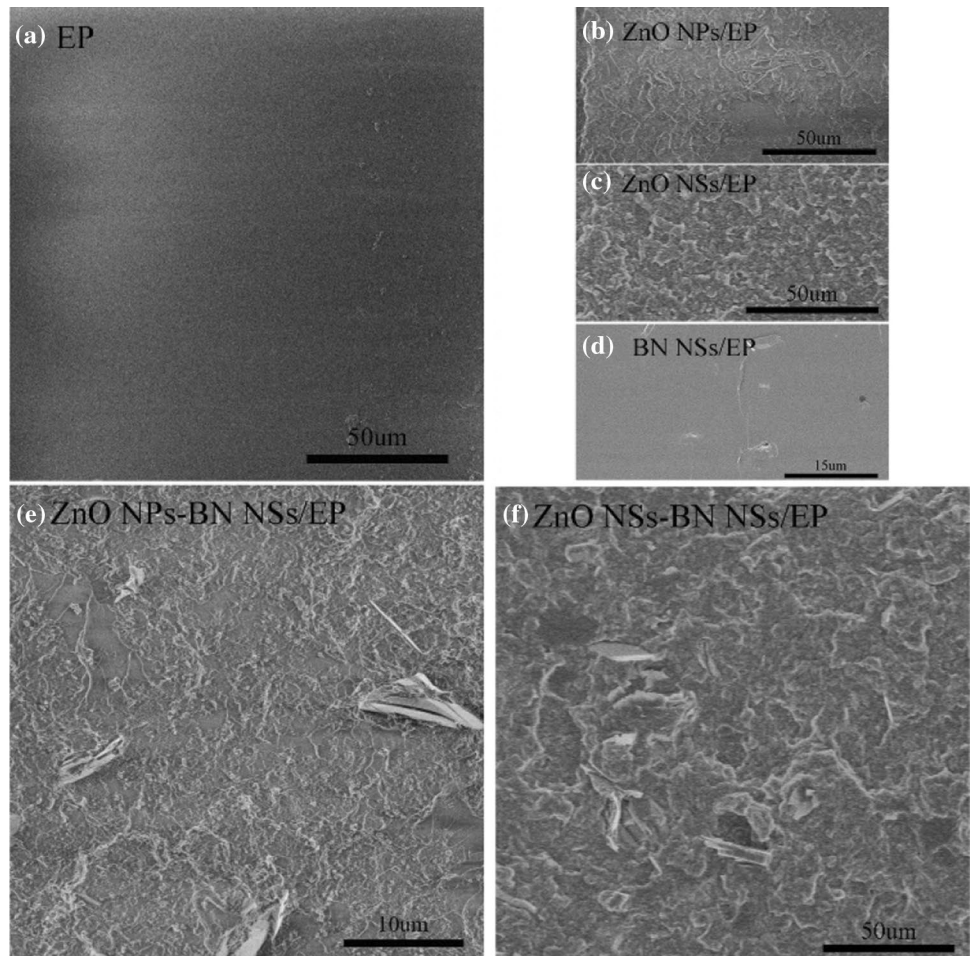


Fig. 4 The relationship between the conductivity and the applied electric field of ZnO/EP composites

The slope of the curve of  $\ln\{-\ln[1-P(E)]\}$  is  $\beta$ , and the intercept is  $E_0$ . The linear fitting results and weibull parameters are shown in Table 1. It can be seen from Table 1 that

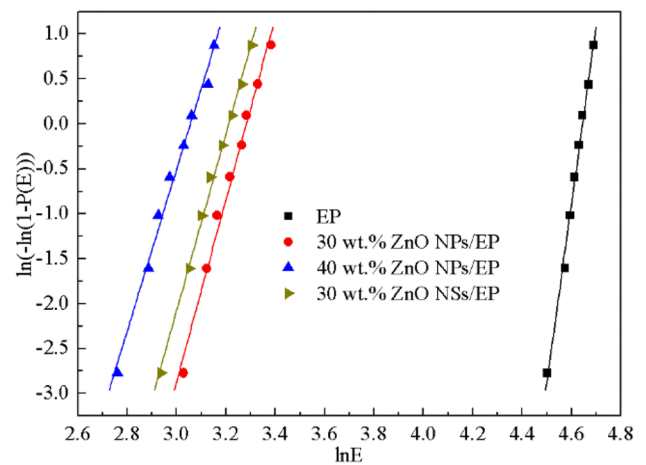


Fig. 5 Weibull curves of ZnO/EP with different shape factors and concentrations

the correlation coefficient  $R$  of all four samples is close to 1, which indicates that all the data have a good linear correlation.  $S$  is the standard error of the least square of the estimation, and a lower  $S$  means a smaller error.

**Table 1** Linear fitting results and weibull parameters of ZnO/EP with different shape factors and concentrations

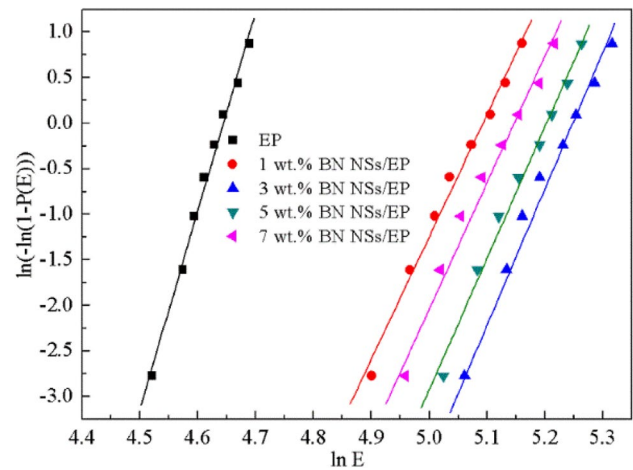
Specimen	Linear fitting results			$S$	Weibull parameters	
	Slope	$\ln(-\ln(1-P(E)))$ intercept	$R$		$\beta$	$E_0$ (kV/mm)
EP	19.86	-92.25	0.99	3.24	19.86	104.06
30 wt% ZnO NPs/EP	10.12	-33.24	0.99	1.38	10.12	26.70
40 wt% ZnONPs/EP	8.99	-27.48	0.99	1.17	8.99	21.26
30 wt% ZnO NSs/EP	9.83	-31.56	0.99	0.79	9.83	24.79

According to Fig. 5 and Table 1, compared with the pure epoxy resin sample, the breakdown strength of the composite sample with different shape factors and different filler loading is significantly reduced. The dc breakdown strength of 30 wt% ZnO NPs/EP, 40 wt% ZnO NPs/EP, and 30 wt% ZnO NSs/EP are 26.70 kV/mm, 21.26 kV/mm, and 24.79 kV/mm, respectively. All of them are only about 25% of the pure epoxy. Although conductivity of the composite has been improved by the single loading semiconductor filler strategy, the dc breakdown performance has been deteriorated seriously. Therefore, both of the electrical performance should be considered at the same time for the safe operation of insulating materials.

In order to solve the problem of insulation deterioration of the composites, two-phase co-doping strategy of insulating filler and semiconductor filler has been proposed. The BN NSs with high thermal conductivity and perfect insulation properties is also widely used to enhance the insulation and thermal management of polymer materials [20, 21]. Therefore, the BN NSs is chosen as another fillers to improve the breakdown strength of ZnO/EP composites. In order to reveal the influence of BN NSs on the breakdown performance of epoxy composites, BN NSs/EP samples with filler loading of 1, 3, 5, and 7 wt% are prepared to select the best component.

In Fig. 6 and Table 2, we can see that the dc breakdown strength of BN NSs/EP nano-composites is much higher than that of pure epoxy, and the 3 wt% has the maximum breakdown strength among all these specimens. The breakdown strength of 3 wt% BN NSs/EP can reach 190.12 kV/mm, which is 82.7% higher than that of pure EP. The improved breakdown strength of EP by filled BN was mainly attributed to the excellent insulating and thermal characteristics of BN NSs. It may be related to the formation of interaction zone around BN NSs.

As the fact that the 3 wt% BN NSs/EP possesses the highest breakdown strength, the co-doped composites of ZnO-3 wt% BN/EP are prepared and the electrical properties are tested. The breakdown strength curves of ZnO-BN/EP composite with different shape factors and different filler loadings are shown in Fig. 7. The linear fitting results and weibull parameters are shown in Table 3.

**Fig. 6** Weibull curves of BN NSs/EP

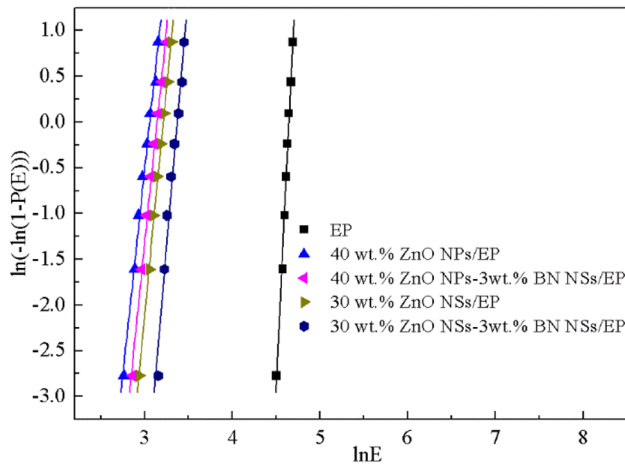
Compared with pure epoxy, the dc breakdown strength of the nonlinear conductive composite material is significantly improved due to the addition of BN sheet. The breakdown strength of 40 wt% ZnO NPs-3 wt% BN NSs/EP, 30 wt% ZnO NSs-3 wt% BN NSs/EP is 23.09 kV/mm and 29.06 kV/mm, respectively, which are increased by 8.6% and 17.2%. The above results show that the breakdown performance of the nonlinear conductive composites can be improved by co-doping ZnO and BN fillers.

The short-time breakdown is mainly related to the density and the mobility of free carriers in the electric field. Base on the multi-region structure model [19, 22], the interaction zone around the nanofiller plays an important role for them. According to the multi-region structure model, the interaction zone consists of a bonded region, a transition region and a normal region. As the filler loading increases, the interaction zone may overlap and the thickness of the interaction zone is enlarged. During the larger thickness of the interaction zone, carriers can gain much more energy from the electric field to transport, and the average density and mobility of carrier increase. The electrical breakdown strength then decreases with the increasing filler loading.

The breakdown mechanism can be further described as shown in Fig. 8. For the composites single doped with ZnO NPs or ZnO Nss, when the filler loading is more then 30

**Table 2** Linear fitting results and weibull parameters of BN NSs/EP

Specimen	Linear fitting results			S	Weibull parameters	
	Slope	$\ln(-\ln(1 - P(E)))$ intercept	R		$\beta$	$E_0$ (kV/mm)
EP	19.86	-92.25	0.99	3.24	19.86	104.06
1 wt%BNSs/EP	13.43	-68.42	0.99	2.87	13.43	163.13
3 wt%BN NSs/EP	14.98	-78.61	0.95	6.41	14.98	190.12
5 wt%BN NSs/EP	14.44	-75.10	0.99	3.19	14.44	181.42
7 wt%BN NSs/EP	13.90	-71.53	0.96	5.59	13.90	171.75



**Fig. 7** Weibull curves of ZnO-BN/EP

wt%, the interaction zone (especially, the transition regions) may connect or overlap. So the carriers may transport along the “easy channel” then the breakdown strength of the composites decreases. For the co-doped composites of ZnO-3 wt% BN/EP, as the introduction of BN sheets, the carrier transport channel is blocked. The carriers have to move through the “hard channels”, then both the density and the mobility of them decrease, so the breakdown strength of the co-doped composites increases.

The nonlinear conductivity of the ZnO-BN/EP composites has been also investigated. The relationship between the conductivity and the applied electric field for

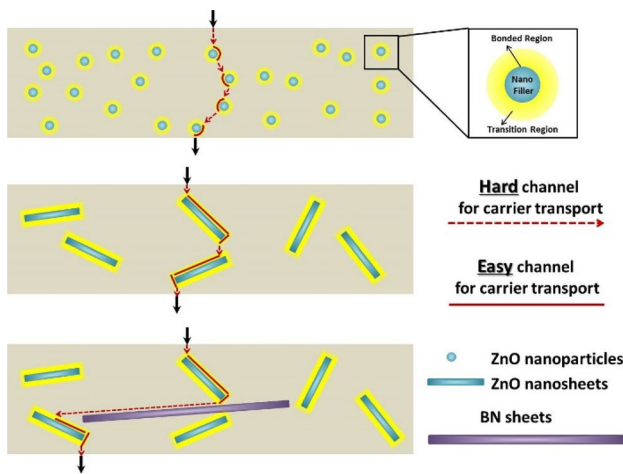
the nano-composites is shown in Fig. 9. Comparing with that of pure EP and ZnO/EP composites, the 40 wt% ZnO NPs-3 wt% BN NSs/EP and 30 wt% ZnO NSs-3 wt% BN NSs/EP also show strong nonlinear conductivity characteristics, and their nonlinear coefficients are 4.36 and 5.05, respectively. Meanwhile, the threshold electric field strength of the co-doped composites are 6.49 kV/mm and 5.51 kV/mm. Compared with the composite sample without BN sheet, the introduction of BN sheet has a small weakening effect on the nonlinear conductivity, but it has a great improvement on the breakdown strength of the nano-composites. Therefore, it can effectively reduce the filler loading, improve the nonlinear conductivity characteristics, and alleviate the deterioration of the breakdown performance of the nano-composites at the same time by regulating the shape factor of the fillers and the co-doping strategy of different types of fillers.

### 4 Conclusions

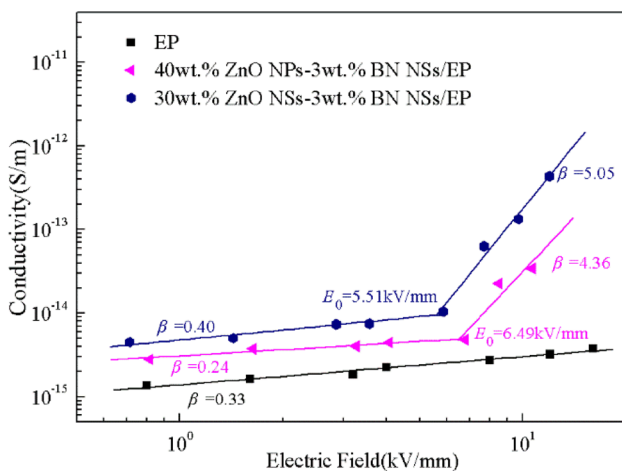
Epoxy resin nano-composites and its electrical properties have been investigated. The epoxy nano-composites are prepared by regulating the shape factor of the semiconductor filling phase, with separate doping or two phase co-doping, and its electrical conductivity and dc breakdown characteristics are tested. The results show that only epoxy composites doped with high filler loading ZnO show obvious nonlinear conductivity. By regulating the shape factor of ZnO, the filler loading and threshold field strength

**Table 3** Linear fitting results and weibull parameters of ZnO-BN/EP

Specimen	Linear fitting results			S	Weibull parameters	
	Slope	$\ln(-\ln(1 - P(E)))$ intercept	R		$\beta$	$E_0$ (kV/mm)
EP	19.86	-92.25	0.99	3.24	19.86	104.06
40 wt% ZnO NPs/EP	8.99	-27.48	0.99	1.17	8.99	21.26
40 wt% ZnO NPs-3 wt% BN NSs/EP	9.41	-29.54	0.99	0.99	9.41	23.09
30 wt% ZnO NSs/EP	9.83	-31.56	0.99	0.79	9.83	24.79
30 wt% ZnO NSs-3 wt% BN NSs/EP	11.56	-38.95	0.96	2.99	11.56	29.06



**Fig. 8** Breakdown mechanism model of epoxy composites



**Fig. 9** The relationship between the conductivity and the applied electric field of ZnO–BN/EP

can be reduced, the nonlinear coefficient can be increased. However, the dc breakdown performance of ZnO/EP is seriously deteriorated, and is only about 25% of the pure EP. The dc breakdown performance of BN NSs/EP is greatly improved, it is 82.7% higher than that of pure EP when the filler loading is 3 wt%. The introduction of BN sheet has a small weakening effect on the nonlinear conductivity, but it has a great improvement on the breakdown strength of the nano-composite. It can effectively reduce the filler loading, improve the nonlinear conductivity characteristics, and alleviate the deterioration of the breakdown performance of the nano-composite by regulating the shape factor of the fillers and the co-doping strategy of different types of fillers. These results may be caused by the interaction zone around nano-particles. The two-phase co-doping nano-composites is beneficial to improve the

electric properties and their applications in the electrical and electronic engineering will be further studied.

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