DC breakdown characteristics of XLPE/BNNS nanocomposites considering BN nanosheet concentration, space charge and temperature

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Abstract: Breakdown failure in insulation material is one of the key problems that threaten the safe operation of high-voltage direct current cable. In this work, the effect of boron nitride nanosheets (BNNSs) concentration, space charge and temperature on DC breakdown strength have been explored. Cross-linked polyethylene (XLPE)/BNNS nanocomposites were prepared by the melt blending method, and the basic characteristics of nanoparticles and composite were characterised. The experimental results indicate that DC breakdown strength of nanocomposite can be effectively improved when a small amount of BN nanosheet is doped into the matrix. The breakdown strength of the sample reaches the maximum value of 407.52 kV/mm when BNNS content is 0.5 wt%, which is about 33% higher than that of pure XLPE. Further, the effect of space charge on the breakdown of nanocomposites has been studied by pre-injecting charges. For the samples with different BNNS contents, all the breakdown strength present ascending trend when the polarity of the applied voltage is the same as that of the pre-injected charges. Besides, it can be found that the breakdown strength of the XLPE/BNNSs composite decreases significantly at 50°C, which is due to more charge accumulation at 50°C. It reaches $2.06 \times 10^{-3}$ C which increases by about 2.2 times than the room temperature.

1 Introduction

Nanodielectric composites have been the focus of research in the field of electrical engineering, since it was firstly proposed by Lewis in 1994 [1]. Cross-linked polyethylene (XLPE) as the main insulation material of high-voltage direct current (HVDC) cable has been widely used due to its excellent electrical insulation properties. However, space charge accumulation is one of the key problems that threaten the safe operation of HVDC cable. Charge accumulation in XLPE will result in local electric field distortion, causing the degradation of the insulation material, even breakdown failures [2–8].

There have been some studies that show the addition of nanoparticles into the polyethylene matrix can effectively inhibit the charge accumulation and improve the breakdown strength of the material [9–13]. Du and co-workers [14] studied the charge transport characteristics of low-density polyethylene(LDPE)/graphene nanocomposites and found that the addition of nanographene can introduce a large number of deep traps, which will inhibit space charge and improve the breakdown strength of nanocomposites. Wang et al. compared the influence of nano-TiO2 on the electrical properties of XLPE before and after modification by the silane coupling agent. They found that the addition of nano-TiO2 can improve the breakdown strength of a composite by 13.5% [15]. Ju et al. [16] studied the effect of modification with different coupling agents on the breakdown strength of MgO/LDPE nanocomposites, and found that the breakdown strength of the modified MgO/LDPE nanocomposite material was significantly higher than that of the unmodified sample.

In the actual application, the insulation materials of HVDC cable are mainly affected by the electric field and thermal field. It is easy to age under high temperature and high voltage, due to the poor thermal conductivity of XLPE matrix, which is the main cause of insulation performance degradation. Hence, improving the thermal conductivity of the insulation material is very important for improving the breakdown strength of the long-term operation of the cable.

In this study, the boron nitride nanosheets (BNNSs) with high thermal conductivity [17–21] have been selected as the filler, which is one of the most thermally conductive materials among many ceramic materials. Hexagonal boron nitride has a layered structure, which is similar to graphite. Unlike graphene, hexagonal boron nitride is a good insulator with high thermal stability, high thermal conductivity and high-temperature insulation [22–26]. Especially, the high thermal conductivity and excellent electrically insulating properties of BNNSs have great potential for fabricating electrically insulating materials. Our previous studies have shown that the addition of BNNSs can effectively inhibit the space charge accumulation in XLPE/BNNSs and improve the insulation performance of the composite [27].

In this work, XLPE/BNNS nanocomposites were prepared by the melt blending method, and the basic characteristics of nanoparticles and composite samples were characterised by Fourier transform infrared spectrum (FTIR), scanning electron microscopy (SEM) and laser flash apparatus. The effect of BNNS concentration on breakdown characteristics of XLPE/BNNSs has been studied. Further, the effect of space charge on breakdown characteristics of XLPE/BNNSs has been studied by pre-injecting charges into the sample. The influence of temperature on the breakdown characteristics of XLPE/BNNSs has been studied. Finally, the above elements on breakdown characteristics are discussed and the breakdown mechanism is analysed.

2 Sample preparation and characterisation

2.1 Surface modification of BN nanosheets

In the experiments, the matrix is a cross-linked polyethylene (XLPE) for HVDC cable insulation, and BNNSs as a modified filler. The dispersion of nanofiller in a polymer matrix directly affects the excellent performance of nanocomposite. In order to realise good dispersion of nanofillers in a polymer matrix, BNNSs were firstly modified with the coupling agent of KH550.

The structure of BNNSs before and after KH550 modification was characterised by FTIR in a wave number range from 400 to...
4000 cm$^{-1}$. Fig. 1 shows the infrared spectra of BNNSs before and after KH550 modification.

It can be seen from Fig. 1 that the absorption peak at 3480 cm$^{-1}$ is strengthened after KH550 modification, corresponding to the stretching vibration peak of $-\text{NH}_2$. The weak absorption peak at 1123 cm$^{-1}$ corresponds to Si–O and the deformation vibration peak of $-\text{NH}_2$ at 1641 cm$^{-1}$. It is certain that KH550 has been successfully grafted onto the surface of BNNSs.

2.2 Preparation of XLPE/BNNS nanocomposites

XLPE/BNNS nanocomposites were prepared by the melt blending method. Prior to the preparation, the treated BNNSs were dried in the vacuum chamber at 60°C for 12 h and XLPE particles were dried at 60°C for 12 h. Firstly, XLPE particles were blended in the internal mixer at 135°C for 5 min and then the desired amount of nanosheets were added to the internal mixer blending for 15 min. Block nanocomposites with different mass fractions were prepared by extruding from the internal mixer, and composite films were prepared by the vulcanising press. Finally, XLPE/BNNS films with six different concentrations, 0.1, 0.5, 1, 3, 5 and 10 wt%, were obtained.

The dispersion of BNNSs in the matrix was observed using SEM. The composite material was firstly quenched in liquid nitrogen and sprayed with a thin gold layer in the section. Fig. 2 shows the SEM images of cross-sections, corresponding to 0.1, 0.5, 3 and 5 wt% XLPE/BNNSs.

It can be seen from Fig. 2 that BNNSs can be observed clearly, and they are dispersed uniformly in the polymer matrix. The thickness of most BNNS changes from 60 to 85 nm, and the average sheet size is about 1 to 3 μm. For high concentration, the amount of BNNSs increases significantly, and there is no obvious agglomeration phenomenon.

2.3 Thermal conductivity characteristics

The thermal conductivity of XLPE/BNNS composites was measured by a laser flash apparatus. The sample thickness is 1 mm and the concentration of BNNSs in the composite is 0, 0.1, 0.5, 1, 3, 5 and 10 wt%. The measured voltage is 260 V and the pulse width is 300 ns. Fig. 3 shows the thermal conductivity of XLPE/BNNS composites.

It can be seen that the thermal conductivity of composites has evidently improved with the increase of BNNS contents. At room temperature, the thermal conductivity of the pure XLPE is 0.328 W/(m K). When the concentration of BNNSs is 10 wt%, the thermal conductivity of the composite material reaches 0.465 W/(m K), increased by about 42% than pure XLPE sample.

It can be explained by the thermally conductive theory [28]. BNNSs with high thermal conductivity are added to the XLPE matrix, which will form the thermally conductive pathways and networks. When BNNS concentration is low, nanosheets were independently and uniformly dispersed in the polymer matrix with little contact with each other, hence the increase of thermal conductivity is relatively small. It increases by about 14.6% for XLPE/BNNS_0.5 wt% than pure XLPE sample. As the concentration of BNNS increases, the distance between the nanometres becomes short, and thermal conduction pathways are extended. It increases by about 42% for XLPE/BNNS_10 wt% than pure XLPE sample. Comparing with the changes in thermal conductivity under high concentration, it increases by about 11% for XLPE/BNNS_10 wt% than XLPE/BNNS_5 wt%. The increase of thermal conductivity is small, which may be due to the fact that when the nano-particles in the matrix reach a certain concentration, similar to the percolation theory, the contribution of nano-particles to thermal conductivity tends to be saturated.

3 Experimental results and analysis

In the experiments, the breakdown strength of XLPE/BNNS nanocomposite was tested by the voltage breakdown test system. In order to study the breakdown characteristics of XLPE/BNNS nanocomposites deeply, three kinds of variables, BNNSs concentration, pre-setting space charge and temperatures, were considered.
which represents the dispersion degree of test results. According to where

\[ E = \frac{F}{\alpha} \]

is the sample number and \( n \) is the sample size.

3.1 Effect of BNNSs concentration on breakdown characteristic

Fig. 4 shows the Weibull probability plot for the breakdown of XLPE and XLPE/BNNS nanocomposite. It can be seen that the breakdown strength of XLPE/BNNSs can be effectively improved when a small amount of BN nanosheet is doped into XLPE. The breakdown strength of the sample reaches the maximum value of 407.52 kV/mm when the BNNS content is 0.5 wt%, which is about 33% higher than that of pure XLPE. Besides, when the concentration is high, the breakdown strength of XLPE/BNNSs reduces instead (Fig. 4).

In order to further analyze the influence of BNNS concentration on the breakdown strength of XLPE/BNNSs, the current of composite with different concentrations was measured by the three-electrode system. It is composed of high-voltage electrodes, test electrodes, ring electrodes and acquisition computers [30]. Prior to the measurement, the specimen was cleaned with anhydrous ethanol and dried in the vacuum drying oven at 60°C. In this experiment, the conduction current of the composite was measured under 20 kV/mm for 15 min. Fig. 5 shows the current of the composite at room temperature.

It can be observed that the current of XLPE/BNNSs shows a rapid decline at the initial time of 60 s. Moreover, the equilibrium value first decreases and then increases with increasing the concentration of nanofiller, which reaches the minimum at a concentration of 0.5 wt%. The variation trend of the composite current with the concentration is consistent with the breakdown experiment results. The breakdown strength reaches the maximum value when the BNNS content is 0.5 wt%.

When the concentration of BNNSs is 0.5 wt%, the interaction zone between the matrix and nanofiller can capture the charge carrier. It inhibits the carrier transport, and significantly enhances the breakdown strength of XLPE/BNNSs. Space charge and trap characteristics of XLPE/BNNSs have been specially studied in our previous work [27]. It indicates that the deep traps [31–33] are introduced between the matrix and nanofiller when a small amount of BNNS is doped into XLPE. The trap levels are 1.15 and 1.02 eV for XLPE/BNNS_0.5 wt% and XLPE/BNNS_1 wt%, respectively. These deep traps near the electrode will capture the injected charges, resulting in charge accumulation near the interface. Further, the local electric field near the interface will weaken the original electric field, thus resulting in a decrease of the injected charges. Hence, the breakdown strength of the nanocomposite can be improved.

When the concentration of BNNSs is high, the interaction zones between matrix and nanofillers may overlap, which will provide a low-resistance path for electrons, and accelerate charge carriers transport through the amorphous zone. It has been studied that shallow traps are introduced owing to physical contact among BNNSs when the concentration of BNNSs is high [27]. The trap level is 0.62 eV and the trap density is \( 5.76 \times 10^{20} \text{ m}^{-3} \) when BNNS concentration is 5 wt%.

3.2 Effect of space charge on breakdown characteristics

In order to further study the influence of space charge on breakdown characteristics of XLPE/BNNSs, some charges are injected into the specimen by applying a certain voltage and then breakdown strength is tested. In the experiment, the specimen was pre-applied electric field under 20 kV/mm for 30 min to accumulate the space charge, and then it was applied voltage with the rising rate of 1 kV/s until the breakdown occurs. Three cases of without pre-applied voltage, pre-applied voltage with the same polarity and pre-applied voltage with the opposite polarity were compared. Fig. 6 demonstrates the Weibull distribution of DC breakdown strength of XLPE/BNNSs after pre-applied voltage.

It can be seen from Fig. 6 that the change of breakdown strength of composites with BNNSs concentration after the pre-applied voltage is consistent with that of without the pre-applied voltage. The breakdown strength of 0.5 wt% is evidently higher than other concentrations. Table 1 shows a comparison of breakdown strength and shape parameter under different pre-applied voltages.
It can be seen from Fig. 6 and Table 1 that the breakdown strength of XLPE/BNNSs composite significantly increases after pre-applied voltage with the same polarity, while it decreases after pre-applied voltage with the opposite polarity. When the specimen was applied electric field of 20 kV/mm for 30 min, some parts of charges will be injected into the specimen, which will affect the breakdown process. It has been measured that the accumulated charge amount of pure XLPE stressed of 20 kV/mm is about 1.08 × 10^{-5} C by pulsed electro-acoustic (PEA) method [27]. The homo-charge near the electrode will weaken the applied electric field, inhibiting the interfacial charge injection, thus improving the breakdown strength. The hetero-polar charges will enhance the applied electric field, resulting in the decline of breakdown strength.

### 3.3 Effect of temperature on breakdown characteristics

In order to study the influence of temperature on the breakdown strength of XLPE/BNNSs composite, four typical temperatures, 25, 50, 70 and 90°C, are considered in the experiments. Fig. 7 shows the breakdown strength of XLPE/BNNSs at 50, 70 and 90°C. Table 2 shows a comparison of breakdown strength and shape parameter at different temperatures.

| Sample, wt% | 0 | 0.1 | 0.5 | 1 | 3 | 5 | 10 |
|-------------|---|-----|-----|---|---|---|----|
| without preloading | α | 306 | 347 | 407 | 373 | 359 | 326 | 266 |
|              | β | 27  | 24  | 21  | 17  | 26  | 25  | 42  |
| homo-polarity | α | 386 | 454 | 492 | 404 | 394 | 355 | 336 |
|              | β | 21  | 70  | 30  | 24  | 23  | 20  | 23  |
| heter-polarity | α | 296 | 331 | 370 | 328 | 320 | 305 | 269 |
|              | β | 19  | 27  | 32  | 30  | 30  | 19  | 19  |

It can be seen that the breakdown strength of XLPE/BNNSs decreases with the increase of temperature. Comparing with room temperature, the breakdown strength of XLPE/BNNSs decreases significantly at 50°C, which is due to the more charge accumulation in the composites at 50°C. The effect of temperature on charge accumulation in XLPE has been specially studied in our previous work [30]. It has been indicated experimentally that there exists an inflection temperature of charge accumulation amount in XLPE, around 50–60°C. The accumulated charge amount reaches 2.06 × 10^{-5} C which increases by about 2.2 times than that of room temperature. These accumulated charges will cause easily local electric field distortion, resulting in local defects inside the material.

When the temperature exceeds around 50°C, the charge conduction will rise markedly, thus the breakdown strength of XLPE/BNNSs has a slight increase at 70°C. However, the breakdown strength of XLPE/BNNSs will evidently decrease at a high temperature of 90°C. It is 258 kV/mm, reducing about 15.7% than that of room temperature. When the temperature increases to 90°C, the external temperature is close to the melting temperature of the XLPE/BNNSs composite. As a result, the material becomes soft, the internal structures are changed and internal defects are increased, which will further reduce the breakdown strength of the composite. Besides, the free electrons gain more energy under the action of high temperatures, intensifying the electron collision ionisation process.

### 3.4 Discussion

The breakdown mechanism of polymer is complex, which can be divided into the intrinsic electrical breakdown, thermal breakdown and electrochemical breakdown and so on [12, 31, 33]. The results show that the space charge effect should be taken into account for DC breakdown in addition to the above mechanism. The space charge accumulation in the polymer will directly affect the breakdown properties. Nano-particles doping can suppress the charge accumulation in the polymer to some extent. Temperature is an important factor affecting the processes of charge injection, migration and accumulation. Fig. 8 shows a logical diagram of BNNS content, temperature and space charge versus breakdown strength.

It can be seen from Fig. 8a a small amount of BNNS doping can significantly improve the breakdown strength of XLPE. Combined with the experimental results in Fig. 4, the breakdown strength of XLPE/BNNS reaches the maximum when the BNNS concentration is 0.5 wt%, increasing by about 33% than pure XLPE. Deep traps in the matrix will be introduced by the doping of BNNS and the injected charges can easily be captured by these deep traps near the electrodes, which will form the local electric field $E_{sc}$. It will weaken the external electric field $E_{app}$, suppressing the further injection of interfacial charges, and the internal charge decreases correspondingly, as shown in Fig. 8d. Generally, the breakdown of the material begins at the ‘electrode–dielectric’ interface. It is concluded that the increase of breakdown strength of XLPE/BNNS is related to the decrease of interface field strength.
which can be verified by breakdown testing after the pre-applied voltage in Fig. 6a. The breakdown strength of XLPE/BNNS significantly enhances after pre-applied voltage with the same polarity, owing to the weakening of interface electric field.

Fig. 8b shows the variation of accumulated charge amount and breakdown strength with temperature. In our previous work [30] the effect of temperature on charge accumulation in XLPE has been specially studied. It has been found that more charges are accumulated in XLPE at 50–60°C than other temperatures. It reaches $2.06 \times 10^{-8} \text{C}$ which increases by about 2.2 times than that of room temperature. In this work, Fig. 7 shows the breakdown strength of XLPE decreases at 50°C than the room temperature, and increases slightly at 70°C. Therefore, it is concluded that the changes in breakdown strength at different temperatures are related to the accumulated charges in the material.

Many studies have shown that nano-particles MgO, TiO$_2$, Al$_2$O$_3$ and so on can suppress the space charge in LDPE or XLPE and enhance the breakdown strength to some extent [9, 16, 32]. In this work, BNNS was selected, mainly considering its good thermal conductivity and unique sheet structure. At present, the maximum operating temperature of HVDC cable is limited to 70°C, which severely limits its conveying capacity. The experiments showed [30] that accumulated charges in XLPE are very small at 90°C, and the main reason for the decline of insulation performance is the damage of heat accumulation to the molecular structure. Therefore, it is key for improving the thermal conductivity of cable material at high temperatures. Fig. 3 preliminarily shows the thermal conductivity property of XLPE/BNNS. In future work, the thermal conductivity of XLPE will specially be studied by means of functionalising nanoparticles or constructing a three-dimensional grid structure. Meanwhile, the electric–thermal breakdown experiment will be carried out.

Table 2  Breakdown strength and shape parameters of XLPE/BNNSs under different temperatures

| Sample, wt% | 0   | 0.1 | 0.5 | 1   | 3   | 5   | 10  |
|-------------|-----|-----|-----|-----|-----|-----|-----|
| 25°C        |     |     |     |     |     |     |     |
| $\alpha$    | 306 | 347 | 407 | 373 | 359 | 326 | 266 |
| $\beta$     | 27  | 24  | 21  | 17  | 26  | 25  | 42  |
| 50°C        |     |     |     |     |     |     |     |
| $\alpha$    | 277 | 333 | 354 | 310 | 269 | 262 | 251 |
| $\beta$     | 18  | 16  | 22  | 29  | 18  | 22  | 35  |
| 70°C        |     |     |     |     |     |     |     |
| $\alpha$    | 299 | 337 | 362 | 332 | 321 | 308 | 262 |
| $\beta$     | 42  | 30  | 34  | 51  | 41  | 24  | 31  |
| 90°C        |     |     |     |     |     |     |     |
| $\alpha$    | 258 | 300 | 334 | 312 | 309 | 280 | 257 |
| $\beta$     | 20  | 26  | 17  | 20  | 21  | 18  | 23  |


4 Conclusion

XLPE/BNNSs nanocomposites have been prepared and characterised. The breakdown characteristics of XLPE/BNNSs nanocomposite have been studied considering the changes of BNNSs concentration, pre-setting space charge and temperature. The conclusions are summarised as follows:

(i) DC breakdown strength of nanocomposite can be effectively improved when a small amount of BN nanosheet is doped into the matrix. The breakdown strength of the sample reaches the maximum value of 407.52 kV/mm when the BNNSs content is 0.5 wt%, which is about 33% higher than that of pure XLPE.

(ii) DC breakdown strength of XLPE/BNNS composite significantly increases after pre-applied voltage with the same polarity, while it decreases after pre-applied voltage with the opposite polarity. The homo-charges near the electrode will weaken the applied electric field, inhibiting the interfacial charge injection, thus improving the breakdown strength. The hetero-polar charges will enhance the applied electric field, resulting in the decline of breakdown strength.

(iii) Compared to room temperature, the breakdown strength of XLPE/BNNSs decreases significantly at 50°C, which is due to more charge accumulation in the composites at 50°C. It reaches 2.06 \times 10^{-5} \text{C} which increases by about 2.2 times than that of room temperature. While the breakdown strength of XLPE/BNNS will evidently decrease at a high temperature of 90°C due to the changes of microstructure under the action of high temperature.

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6 References

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