

# The Application of Modified Graphene with KH570 Used in Semiconductive Shielding Material of Power Cable

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**Abstract:** In order to reduce the content of conductive filler and improve the smoothness of semiconductive shielding layer of power cable, Graphene was used as a better kind of conductive filler to replace a part of carbon black (CB). In the meantime, in order to improve the consistency between the graphene and ethylene-vinyl acetate copolymer (EVA),  $\gamma$ -methacryloxypropyl trimethoxy silane (KH570) was used to modify the graphene. The result shows that modified reduced graphene oxide (MrGO) has a better compatibility and dispersability with EVA matrix. A qualified composite can be obtained only by adding 6% of MrGO and 10% of CB, compared with traditional semiconductive shielding material, the conductive filler content of which has been reduced greatly. In addition, this kind of new material has a good mechanical, electrical, thermal properties.

**Keywords:** Modified Grapheme, Semiconductive Shielding Material, KH570, EVA

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## 1. Introduction

Recently, the application of power cable with shielding layer in power transmission system is more and more popular. Shielding layer was mainly used to provide a smooth interface between conductor and insulation layer to protect the insulation from partial discharges caused by conductor [1]. The service life of cable is related to the smoothness degree of shielding layer significantly. Generally, the preparation process of shielding material of crosslinked polyethylene (XLPE) cable mainly adopts adding CB into EVA matrix to obtain a conductive composite. Addition amount of CB varies from 15wt% to 50wt% to reduce volume resistivity of shielding material lower than 100  $\Omega \cdot \text{cm}$  [2]. However, considering the smoothness and extrusion behaviour of shielding material simultaneously, the amount of CB is inappropriate to add too much [3]. Therefore, reducing the amount of conductive filler is very important.

As a new kind of conductive material, graphene has excellent behaviour in thermal and mechanical properties, which makes it possible to use in the shielding material.

However, its high chemical stability and inert surface, which can decrease the mechanical property of composite strongly [4], makes it not easy to combine with EVA matrix consistently. Moreover, its bad dispersibility, resulting from aggregating easily [5], also makes it difficult to apply in industry. Therefore, the modification of graphene becomes a necessary procedure for its large-scaled application in polymers. For example, Kotal et al. [6] reported that they have modified graphene by pphenylenediamine (PPD), this kind of modified graphene shows intimate interaction with bromo butyl rubber (BIIR) and can be dispersed very well. Fu et al. [7] have synthesized an amine-functionalized nanosilicagrafted graphene (OAS-graphene). This modification process not only helped graphene dispersion in polymer but also provide the  $-\text{NH}_2$  active sites linking the graphene and the epoxy matrix, which improved the interface quality effectively. As a kind of silane coupling agent, KH570 is often used for modifying inorganic filler in order to improve its consistency and dispersibility in polymer material [8]. KH570 modified GO (MGO) can be prepared based on trialkoxysilanes of KH570 molecules firstly hydrolyse and then condense with hydroxyls

on the surface of graphene oxide (GO). After reduction as MrGO, the functional groups of which can react with double bond of unsaturated polyester so that it can be combined with polymer tightly.

In this paper, MrGO was prepared and characterized by FT-IR, XRD, Raman. Then EVA/CB-MrGO composite materials were studied, and its electrical conductivity, mechanical property and morphology were tested if it is suitable using in the shielding layer of power cable.

## 2. Experimental

### 2.1. Materials

GO and ordinary graphene nanoplate (GNS) was purchased from Nanjing XFNANO Materials Tech Co., Ltd; KH570 was purchased from Shandong Xiya Chemistry Co., Ltd; EVA was supplied by DuPont, the mark is EVA260.

### 2.2. Sample Preparation

#### 2.2.1. Preparation of Modified Graphene Oxide

Firstly, 1 g of GO was dispersed in 600 ml EtOH by 0.5h ultrasonic treatment. Secondly,  $\text{CH}_3\text{COOH}$  was added into GO dispersion to adjust pH at 3-4. In the meantime, 3 g of KH570 was mixed with deionized water and EtOH (KH570: deionized water: EtOH = 1: 1: 20 in weigh), and then after 15min hydrolyzation at room temperature, KH570 solution was injected into acid GO dispersion slowly with stirring. The mixture was allowed to react at 70°C for 24h and then isolated by centrifugation. The precipitate from the centrifugation was washed in succession with 120 ml EtOH and 120 ml deionized water. Finally, MGO solid was obtained after drying in vacuum oven at 60°C for 12h.

MrGO was prepared by reducing MGO at hydriodic acid with acetic acid (HI-AcOH) system [9]. Firstly, 1 g of MGO was dispersed in 0.5 L AcOH. Following, 20 ml HI was added. After that, the mixture was kept reacting at 50°C for 24h with constant stirring. When reaction finished, the mixture was treated by centrifugation isolation followed with 120 ml EtOH, 120 ml 30% HCl and 120 ml deionized water washes respectively. Finally, MrGO solid was obtained by vacuum-dried at 60°C for 12h.

#### 2.2.2. Fabrication of Composite Material

In this work, EVA/CB-MrGO, EVA/CB-GNS and EVA/CB composites were fabricated. EVA/CB-MrGO composites was prepared via solvent-blending procedure as follows: different content of MrGO (3wt%, 4wt%, 5wt%, 6wt%, 7wt%, which account for the total weigh of EVA) was dispersed in THF (2 mg/ml) with ultrasonic treatment for 0.5h. Subsequently, 10wt% of CB was added under continuous stirring for 10min in the resulting MrGO dispersion to form CB/MrGO dispersion. Subsequently, 5 g of EVA was added into the mixture of dispersed CB/MrGO and the mixture was stirred at 70°C for 30min. Then the solution of EVA/CB-MrGO was vacuum-dried at 40°C for 5h to obtain EVA/CB-MrGO composites.

EVA/CB-GNS composite was also prepared by using the

same way described above. The only difference was that the MrGO was changed into GNS.

For EVA/CB composite, 10wt% of CB was dispersed in THF (2 mg/ml). Then 5 g of EVA was added and the mixture was under continuous stirring at 70°C for 30min. Then the solution of EVA/CB was vacuum-dried at 40°C for 5h to obtain EVA/CB composite.

Finally, three kinds of composites were compression moulded into films by press vulcanizer at 120 °C for 5min, under 5MPa.

### 2.3. Characterization

The structures of GO, MGO and MrGO were characterized by Fourier transform Infrared Spectrometer (FT-IR, Bruker EQUINOX 55) with range of wavelength from 4000  $\text{cm}^{-1}$  to 400  $\text{cm}^{-1}$ . The samples were prepared with KBr.

The melting and crystallization behavior were investigated by Drilling String Compensator (DSC, TA Q20). Samples were heated from -30 °C to 150 °C at 10 °C /min to eliminate thermal history, then cooled to -30 °C at the same rate to analyse their crystallization property and heated again to analyse their melting property.

The morphology analysis was characterized by transmission electron micrograph (TEM, JEOL JEM-2100F)

The diffraction behavior of GO, MGO and MrGO were studied by X-ray diffractometer (Bruker D8 Advance), using Cu  $\text{K}\alpha$  radiation ( $\lambda=0.154\text{nm}$ ) with voltage of 40kV and a current of 40mA at a scanning rate of 2°/min.

The structure of MGO and MrGO were characterized by raman spectrometer (thermo scientific DXR) with 532nm excitation wavelength.

Fracture surfaces of the composite samples were characterized by scanning electron microscope (SEM JEOL JSM-6360LV).

The electrical property of composite samples was tested using two-probe test fixture (ST2258C Suzhou Jingge Electronic) at 23°C, 50% of relative humidity based on National Standard of China GB/T 3048.3-1994.

The mechanical property of composite samples was tested by electronic tensile testing machine (CMT4304 Shenzhen Xinsansi Marerial) was performed followed the National Standard of China GB/T 1040-1992 at a strain rate of 20 mm/min.

### 2.4. Measurement of Dispersion Stability

The dispersion stabilities of MrGO and ordinary graphene were researched by testing their sedimentation volumes. 0.05 g of samples were dispersed in the THF by 0.5h ultrasonic treatment in a 10 ml graduated cylinder. After a period of time, the suspension began to sedimentate and divide into THF supernate and sediment. Sedimentation volume was recorded as V every 5min.

## 3. Results and Discussion

### 3.1. FT-IR Analysis

Figure 1 shows the FT-IR spectra of GO, MGO, MrGO. The

broad peak at  $3420\text{ cm}^{-1}$ , apparent at three spectra, ascribes to the stretching vibration of O-H groups from GO and absorbed water molecules. Other oxygen containing functional groups can be observed at the spectrum of GO such as alkoxy C-O ( $1050\text{ cm}^{-1}$ ), epoxy C-O ( $1223\text{ cm}^{-1}$ ) and C=O ( $1720\text{ cm}^{-1}$ ). And the peak at  $1625\text{ cm}^{-1}$  is related to C=C/ C-C stretching [10, 11]. The new peaks from at  $2878\text{ cm}^{-1}$  and  $2924\text{ cm}^{-1}$  from MGO are contributed to the methyl and methylene come from KH570 respectively. The peaks at  $1050\text{ cm}^{-1}$  and  $1223\text{ cm}^{-1}$  disappeared, which is replaced by a new peak at  $1080\text{ cm}^{-1}$ . It arises from Si-O-Si/ Si-O-C formed by reaction between the hydroxide radical on the surface of GO and a part of silanol which comes from the hydrolysis of Si(OCH<sub>3</sub>) in KH570 (Figure 2). It is demonstrated that KH570 has been grafted onto the surface of GO successfully. After being reduced via HI, there is a peak at  $1640\text{ cm}^{-1}$  which is the characteristic peak of graphene, indicating that MGO has been reduced to MrGO. Meanwhile, the intensities of oxygen containing functional groups decreases dramatically also proves it. On the other hand, the peaks of methyl and methylene at  $2878\text{ cm}^{-1}$  and  $2924\text{ cm}^{-1}$  remain existed. It means KH570 still combined with GO tightly. There is a broader peak from  $1303\text{ cm}^{-1}$  to  $1020\text{ cm}^{-1}$ . It may because there are more Si-O-Si were obtained by the hydrolytic condensation of Si(OCH<sub>3</sub>) in the process of reduction which were loaded at the surface of GO [12].

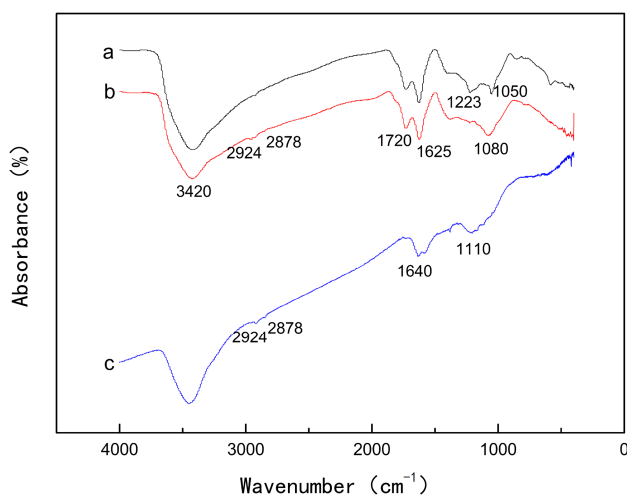


Figure 1. FT-IR spectra of (a) GO (b) MGO (c) MrGO.

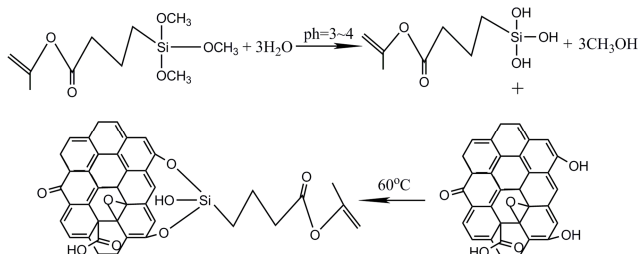


Figure 2. Hydrolysis reaction of KH570 and modification mechanism of GO.

### 3.2. XRD Analysis

Figure 3 presents the XRD spectra of GO, MGO, MrGO. There is a diffraction peak (001) of GO at  $10.52^\circ$ , while after

modified, the peak of MGO is detected on the lower angle located at  $10.39^\circ$  and is becoming sharper and stronger. It means the interlayer spacing of MGO becomes larger from  $0.84\text{ nm}$  to  $0.851\text{ nm}$ . That's because the functional groups from KH570 insert into the layers of GO. The two new broad peaks appear at  $24.19^\circ$  and  $42.99^\circ$  on the XRD spectra of MrGO, are an indication of the reduction of MGO, corresponding diffraction peak (002) and diffraction peak (001) of MrGO respectively [13].

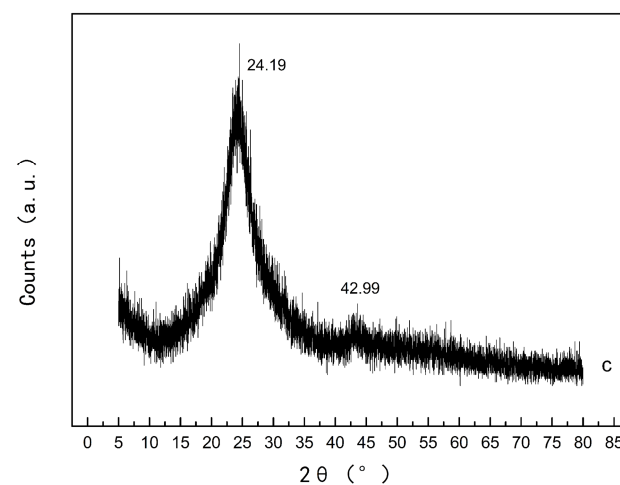
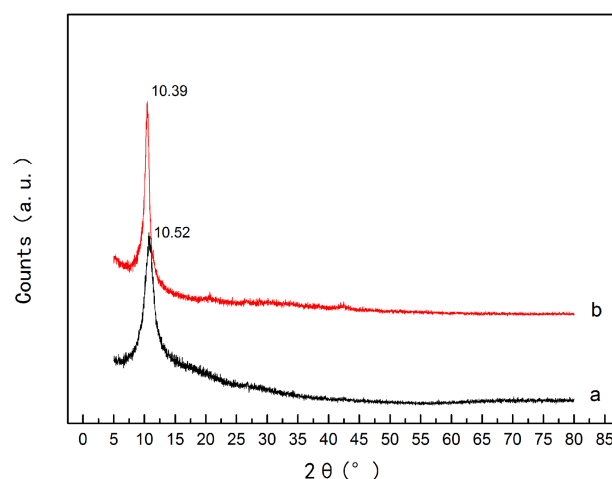


Figure 3. The XRD of (a) graphene oxide (b) modified graphene oxide (c) modified grapheme.

### 3.3. Raman Spectrum

Reduction degree of MGO and structural defects of MrGO can be detected by analysing the location and intensity of D-band and G-band in Raman spectrum. In Figure 4, it is shown that the peaks of G for MGO is located at  $\sim 1590\text{ cm}^{-1}$ , which is higher than that of graphite ( $\sim 1581\text{ cm}^{-1}$ ) has been reported before [14]. The up-shifted of G-band is because the presence of isolated double bonds that separated by functional groups of MGO [15].

The D-band and G-band position of MrGO are  $\sim 1370\text{ cm}^{-1}$  and  $\sim 1590\text{ cm}^{-1}$  respectively. Compared to MGO, the ratio of intensity of D peak and G peak increased markedly. The

variation is contributed to the presence of more defect concentration after the MGO was reduced [16].

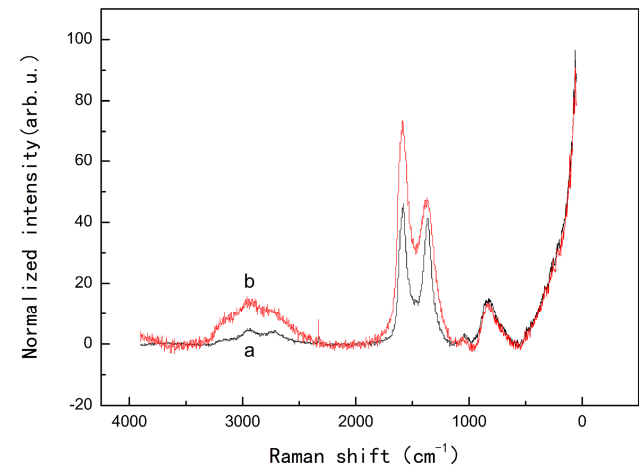


Figure 4. The Raman of (a) MGO (b) MrGO.

3.4. TEM Analysis

The TEM images of MrGO and ordinary graphene are shown in Figure 5. compared to the ordinary graphene, the surface area of MrGO decreased significantly that are benefit to the dispersion in polymer [17]. Moreover, MrGO becomes infolded. It maybe owing to minimization of free energy after grafting KH570 [18]. Based on the same quality of ordinary graphene and MrGO, it is easier to construct a three-dimensional conductive network when MrGO is used in the shielding material because more CB can be loaded on the infolded surface of MrGO, like the TEM micrograph showed in Figure 6.

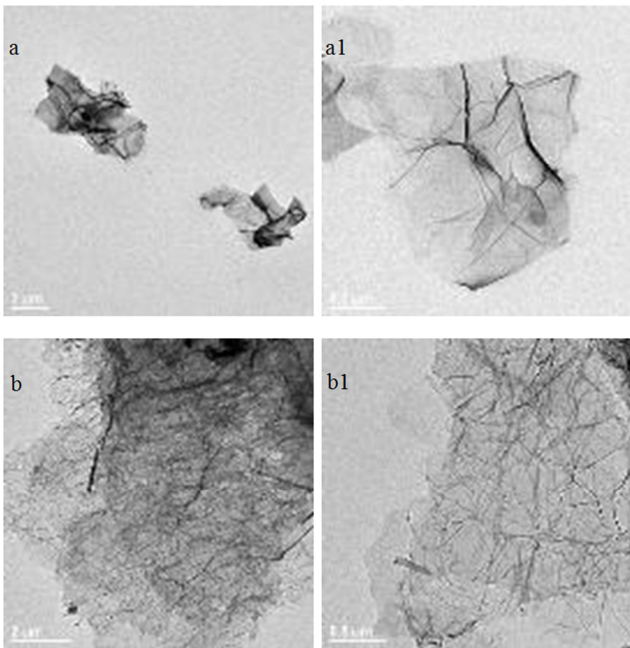


Figure 5. TEM images of (a) MrGO (b) ordinary graphene.

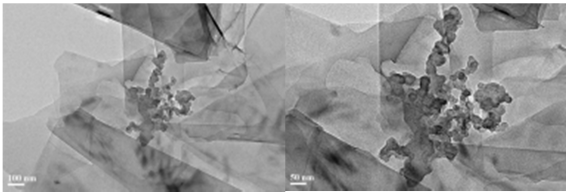


Figure 6. TEM micrograph of MrGO and CB.

3.5. Dispersion Stability

MrGO has a good compatibility with EVA matrix owing to its strong lipophilic degree. Figure 7 shows 10min dispersion state of MrGO in the deionized water and THF with 5 mg/ml concentration after 0.5h ultrasonic treatment. It can be seen that MrGO can't be dispersed in deionized water while it can be dispersed in THF stably. That's because the MrGO surface is grafted by KH570, a kind of hydrophobic group that can help it to disperse in organic solvent.

Filler dispersion stability in solvent is important when using mixed solution method to manufacture composite material. It is primary work for the next mixing step. The sedimentation volume comparison between MrGO and ordinary graphene in THF was constructed in Figure 8. It can be observed that sedimentation velocity of MrGO is slower than that of ordinary graphene in the first 30min, which means MrGO has a better dispersability in THF.

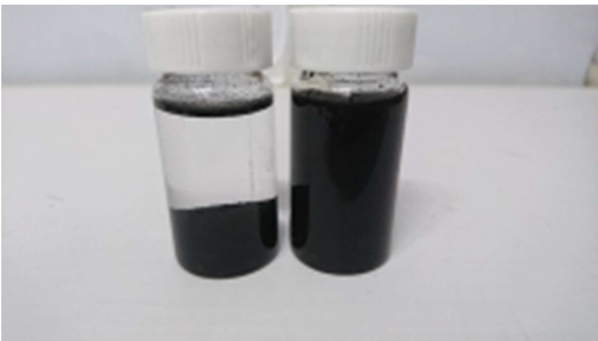


Figure 7. Dispersion stability in deionized water(left) and THF(right).

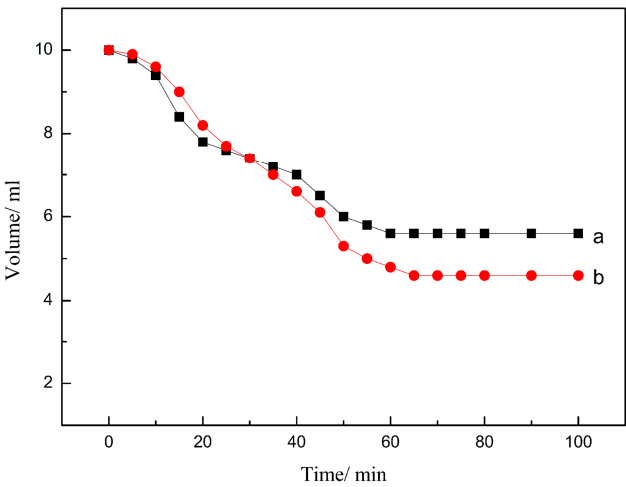


Figure 8. Dispersion stability of (a) ordinary graphene (b) MrGO in THF.



### 3.6. Electrical Property of Composite

A good conductive performance is the most important standard of shielding materials. According to National Machinery Industrial Standard of China JB/T-10738-2007,

the volume resistivity of semiconductive shielding material for cross-linked polyethylene insulated cable must be lower than  $100 \Omega \cdot \text{cm}$ . The volume resistivity of EVA/10%CB-MrGO composites are shown in table 1.

**Table 1.** Volume resistivity of EVA/10%CB-MrGO filled with different content of MrGO.

Content of MrGO/%	3.0	4.0	5.0	6.0	7.0
Volume resistivity/ $\Omega \cdot \text{cm}$	$5.4 \times 10^3$	$2.4 \times 10^3$	210	92.2	56.9

From Table 1, when the content of CB was confirmed to 10%, it could be found that the volume resistivity of composite was decreased with an increase in the content of MrGO. Moreover, there was a sharp decrease in volume resistivity with an increase loading of MrGO from 3% to 5%, with volume resistivity dropping from  $5.4 \times 10^3 \Omega \cdot \text{cm}$  to  $210 \Omega \cdot \text{cm}$ . when continue increasing the content of MrGO, the increasing range of volume resistivity was not as apparent as former. This phenomenon was consistent with the literature reported before [3]. When the content of MrGO increased to 6%, the volume resistivity of composite was reduced lower than  $100 \Omega \cdot \text{cm}$ .

### 3.7. Mechanical Property of Composite

KH570 was always be used as coupling agents to enhance interaction between organic and inorganic phases [19]. On the other hand, it is also used to help improve dispersion ability of inorganic filler in the polymer [20]. The SEM micrograph images of EVA/CB, EVA/CB-GNS, EVA/CB-MrGO composites are shown in Figure 9.

Compared to Figure 9(a, a1), it is obvious in (b, c) that the surface of EVA/10%CB-GNS turned irregularity after adding ordinary graphene because of their bad compatibility, which is not benefit to the mechanical property of composite. However, the surface of EVA/10%CB-MrGO is more smooth, the morphology of EVA didn't destroy after adding MrGO. It does prove MrGO and EVA matrix have good surface interactions. This could be attributed to the improved affinity between the acrylate groups from KH570 and vinyl acetate from EVA. Furthermore, CB shows a poor dispersability in Figure 7(a), but it's better in Figure 7(d, e). It may because CB particles were loaded on the surface of MrGO uniformly rather than distributed randomly inside the EVA matrix. Another advantage is a three-dimensional conductive network can be formed due to the synergistic effect between CB and MrGO [21]. According to National Machinery Industrial Standard of China JB/T 10738-2007, the elongation at break and tensile strength of semiconductive shielding material for cross-linked polyethylene insulated cable must be higher than 200% and 10MPa, respectively. Table 2 shows the mechanical property of composite changing with the content of MrGO.

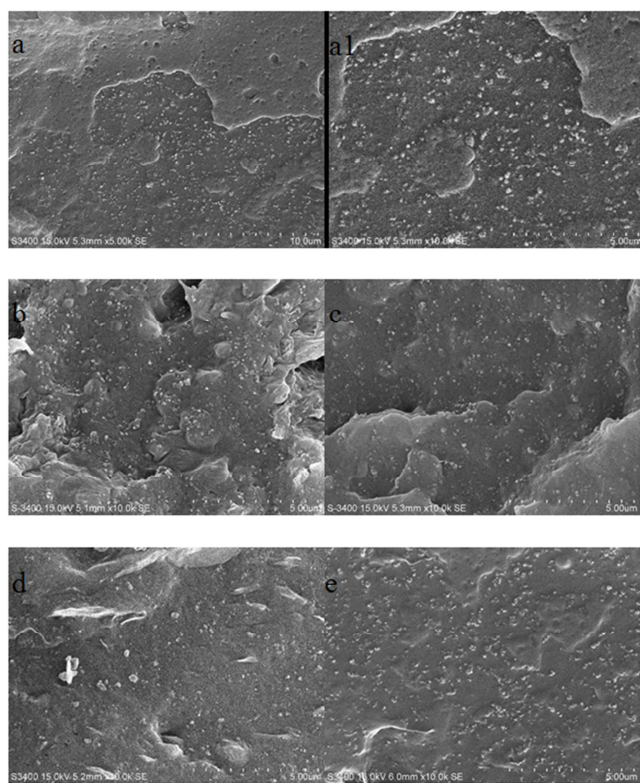
**Table 2.** Effect of the content of MrGO on mechanical property of EVA/10%CB-MrGO composites.

Content of MrGO/%	0	3	4	5	6	7
Elongation at break /%	703.2	651.4	623.6	580.9	554.1	489
Elasticity modulus /MPa	17.7	20.9	22.5	22	22.7	21.3
Tensile strength /MPa	19.8	17.5	16.1	14.2	13.8	12.9

From Table 2, its elongation at break and tensile strength both decreased when the content of MrGO is increasing. Though both of them are higher than industrial standard. But the elasticity modulus is increasing because of the excellent elasticity modulus of MrGO [22].

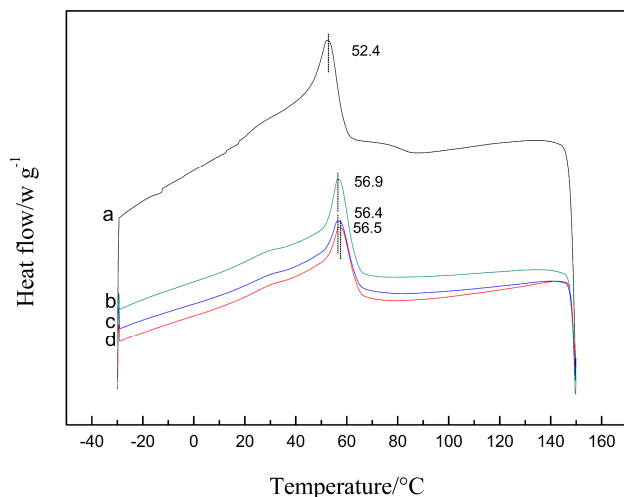
### 3.8. Thermal Property of Composites

The Figure 10 of DSC shows the cooling curves of composites. It can be seen that the location of crystallization peak increased from  $52.4^\circ\text{C}$  to  $56.9^\circ\text{C}$  after adding only 3% of MrGO implies that the MrGO results in a higher crystallinity than the composite without MrGO. MrGO has played a heterogeneous nucleus role in the EVA matrix that enhanced the crystallization rate of composite [23]. Whereas the location of crystallization peak almost unchanged with increasing MrGO contents. The heterogeneous nucleation effect of MrGO became less obvious that may because the movement of polymer molecular chains are blocked by the



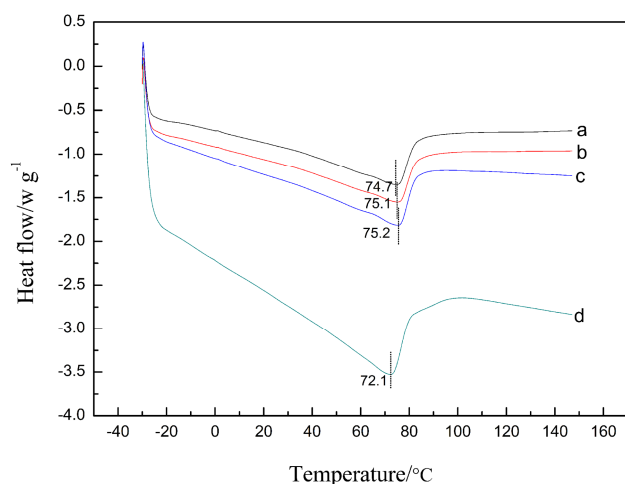
**Figure 9.** SEM micrograph images of the (a, a1) EVA/10%CB (b) EVA/10%CB-5%GNS (c) EVA/10%CB-3%GNS (d) EVA/10%CB-5%MrGO (e) EVA/10%CB-3%MrGO.

high addition content of MrGO, and there is more contact probability between EVA and MrGO, which might reduce the effect of crystal lattice [24]. In general, the crystallization behaviour depends on the MrGO impact on both heterogeneous nucleation and crystal growth.



**Figure 10.** The cooling thermograms of composites (a) EVA/10%CB (b-d) EVA/10%CB-MrGO, the content of MrGO is 3%, 5%, 7% respectively.

The Figure 11 of DSC shows the melting behaviour of composites. It is obvious that the position of melting peak moves toward high temperature after adding 3wt% of MrGO. To some degree, the melting peak can reflect the crystallization conditions [25]. This indicates that the presence of the MrGO increases the crystal size and its perfection. Nevertheless, this phenomenon isn't apparent with continuous increasing of MrGO that is coincident with the analysis before.



**Figure 11.** The heating thermograms of composites (a-c) EVA/10%CB-MrGO, the content of MrGO is 3%, 5%, 7% respectively (d) EVA/10%CB.

## 4. Conclusion

In conclusion, there is a high application value for MrGO as conductive filler using in the semiconductive shielding

material. XRD, FT-IR and Raman analyses of MGO and MrGO powders clearly demonstrated that the KH570 was grafted onto the surface of GO successfully. After modified by KH570, MrGO can be dispersed into THF more uniform than ordinary graphene so that it can be mixed with EVA solution more uniform in the next step. The SEM of composite material proves that MrGO has a good compatibility and dispersability with EVA matrix.

The EVA/CB-MrGO was prepared by mixing two kinds of conductive fillers, CB and MrGO, into EVA matrix. This kind of new material has a good mechanical, electrical properties when the contents of CB and MrGO are confirmed as 10% and 6%. Moreover, The addition of MrGO favors the crystallization behaviour of the composite.

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## Biography



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