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A large Improvement in Thermal Conductivity and Anti-static Property of Epoxy Resin by Doping Graphene

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ABSTRACT

Facing the increasing integration and miniaturization of electronic devices, effective thermal dissipation has become a crucial factor in ensuring the safety and reliability of electronic devices. Based on its extraordinary thermal conductive properties, graphene has become one of the most promising material regarding heat dissipation issues. In this work, graphene fillers were embedded into the epoxy resin to prepare a material that can be used in the encapsulation field for electric devices. The function of fillers on the thermal conductivity and the thermal stability of samples is studied. Partial discharges are tested and the anti-static properties of the samples were investigated by surface charge decay property. It is found that the thermal conductivity of sample with 0.1 wt% loading of graphene is increased about 1.6 times. Meanwhile, the thermal stability and the resistance of anti-static of graphene modified samples are both improved. The findings in this paper provides a reference to develop a variety of outstanding thermal conductivity materials and approve the practicability of graphene/EP for using in the encapsulation filed of electronic devices.

Index Terms — Epoxy resin; graphene; surface charge; thermal conductivity; PD

1 INTRODUCTION

WITH the rapid development of science and technology, the electronic device and its circuits are becoming more miniaturization and high integration in a wide range of fields including aerospace, diving, military, energy, etc. [1,2]. With the significant improvement of the performance for these devices, the power consumption and heat releasing are increasing as a consequence during operation [3]. The heat generated in the system can be kept by the capsuled dielectrics, which affects the efficiency, stability and lifetime of these electronic devices.

Meanwhile, the electrostatic charge generated on the dielectric surface can probably aging the local insulation and even endanger the safe operation of the system. Therefore, the effective diffusion and anti-static property of dielectrics for electronic devices has become one issue that desperately needs to be solved in the development of modern electronic devices [4].

Due to its outstanding mechanical stiffness, chemical resistance, and superior adhesion, etc., epoxy resin (EP) is extensively applied to a wide range of important fields as encapsulating material [9]. However, the intrinsic properties regarding thermal conductivity, thermal stability, and anti-static of EP brought major disadvantages as the application of surface coatings or encapsulating materials for electronic devices [10,11].

Graphene, a monolayer of carbon atoms arranged in a two-dimensional honeycomb lattice, has attracted extensive attention

in energy storage, sensor and thermal interface material fields due to its excellent thermal conductivity, electrical properties, chemical stability, flexibility and mechanical strength [12-14]. It was found by Liu et al. that with the increase of graphene content in EP, a significant increase of the thermal conductivity and flame retardancy for composites was obtained [15]. Song et al. fabricated a functional graphene with lower defect and synthesized EP composites. Their result showed that the functional graphene was easier to dispersion in matrix which results in an increase in the thermal conductivity [16]. Fu et al. studied the thermal properties of EP doped with several representative fillers with better thermal conductivities and found that the addition of fillers not only enhanced the thermal conductivities of composites, but also improved the thermal stability [5]. Tang et al. proved in his work that the graphene/EP can have a thermal conductivity increasing of about 4.9 W/m.K with a filler loading fraction of 30wt%, which was about the 19 times higher compared to that of the neat EP.

Based on the reviews, it can be concluded that doping of graphene can be useful to tailor thermal property of dielectric materials. However, during the operation of electronic devices, electrostatic charging phenomenon often occurs, which could likely induce the partial discharge (PD) and result in a degradation of materials. Very few previous researches investigated the electrical properties of composites as encapsulation materials.

In this work, EP was doped with graphene with a mass ratio of 0.1 wt% to fabricate the thermal dissipation composites for electronic devices. The thermal property and electrical properties were investigated. It is found that compared with the neat EP, the addition of graphene can enhance the thermal conductivity of EP greatly. More importantly, the resistance of PD activity and improvement of anti-static properties implies a potential application to extending the service life of electronic devices.

2 EXPERIMENT SETUP

2.1 EXPERIMENTAL SAMPLES

The epoxy resin (E-51, epoxide equivalent 184-195 g/mol) was purchased from Usoft chemical Co., Ltd., Shandong, China. The poly(propylene glycol) bis(2-aminopropyl) ether (D230, density 0.93-0.97g/cm³) as a curing agent was purchased from Haozhuo material technology Co., Ltd., Nanjing, China. The graphene was supplied by the Institute of Semiconductors, Chinese Academy of Sciences. The absolute ethyl alcohol was purchased from Damao chemical reagent factory, Tianjin, China. All the materials were used without further purification.

Disperse the graphene in absolute ethyl alcohol with ultrasonic treatment for 0.5 h, before adding to the epoxy resin and stirring for 2h at 60°C. The whole process was carried out under the heating condition to make the graphene fully and uniformly dispersed in the epoxy system. Add the curing agent to the mixture system with the mass ratio of epoxy and curing agent being 3:1 and vigorous stirring for 1 hours at 60°C. Then, the mixed solution was poured into a stainless steel mold, precured in an oven at 80°C for 2h, and postcured at 120°C for 2h to obtain the graphene/EP composite. The fabrication process of graphene/EP

composites was shown in Figure 1. The thickness of the samples was 0.5 mm.

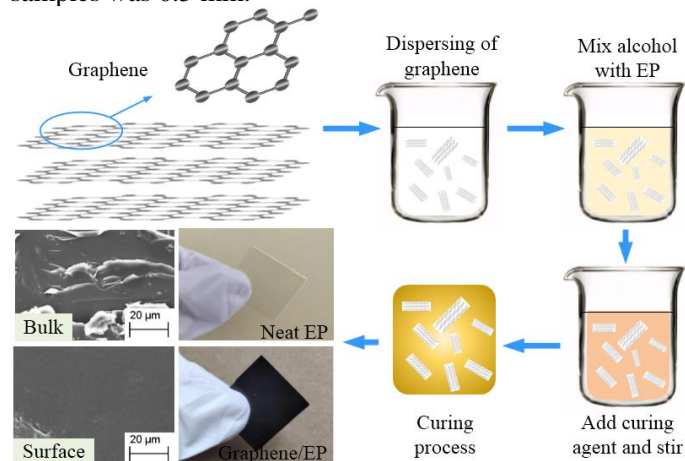


Figure 1. The fabrication process of graphene/EP composites.

2.2 CHARACTERIZATION

The thermodynamic change of materials was measured by differential scanning calorimetry (DSC) analyzer (DSC 131 Evo, SETARAM, France). During the measurement, the hollow crucible was used as the reference, with an increasing rate of 10 °C/min during the heating process till the temperature reaches 300 °C, and with a decreasing rate of 20 °C/min during the cooling process till temperature reach 40 °C. Repeat the heating process and the scan curve can manifest the glass transition temperature (T_g) of samples. Thermal conductivities of samples were measured by the heat flow DRL-III high precision thermal conductivity meter (Xiangtan Instrument and Meter Co., Ltd. Hunan, China) with measurement accuracy higher than 3%. The parameters and method used in this experiment were conformed based on the ASTM-D5470-12.

A rod-plate electrode system was used and the PD tests were conducted under sinusoidal voltage whose amplitude and frequency were 1.5 kV and 50 Hz, respectively.

Meanwhile, in the operation of electronic devices, the static charge maybe cause imponderable harm to the system. Therefore, it is important to verify the antistatic property of the modified material. According to the IEC 61340-2-1, the material is charged by a corona discharge of -10kV for 10 minutes using a needle electrode. The distance between the needle electrode and the sample was 10 mm. Before discharge process, samples (40mm×40mm×0.5mm) were cleaned by absolute ethyl alcohol, and dried by the ionizing blower to eliminate the interference of surface static charges. The corona discharge set-up can be found in our previous articles [a, b].

3 RESULTS

3.1 PHYSICAL PROPERTIES

It is shown in Figure 2 that the contact angle of EP is about 88°, while the contact angle of graphene/EP is increased up to around 102°. The results indicate that the addition of graphene can enhance the hydrophobicity of EP. The contact angle is dependent on the surface morphology and its intrinsic properties of the compositional constituents [20]. The hydrophobic characteristic can effective limit the water or

corrosive solution adsorption on the surface of malt is shown in Figure 2 that the contact angle of EP is about 88° , while the contact angle of graphene/EP is increased up to around 102° . The results indicate that the addition of graphene can enhance the hydrophobicity of EP. The contact angle is dependent on the surface morphology and its intrinsic properties of the compositional constituents[20]. The hydrophobic characteristic can effectively limit the water or corrosive solution adsorption on the surface of materials that can ensure the safe and efficient operation of equipment and guarantee its service lifespan.

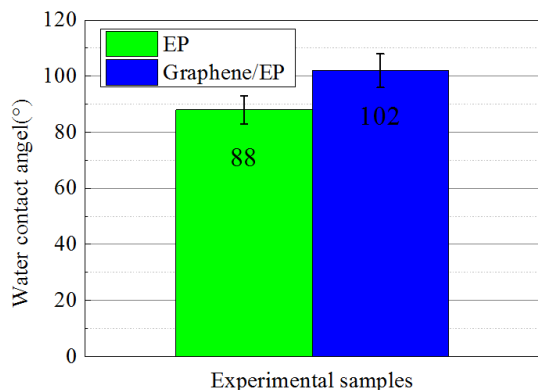


Figure 2. The water contact angle of EP and graphene/EP.

3.2 THERMAL PROPERTIES

The glass transition temperature test result of experimental samples is shown in Figure 3. It can be seen that the glass transition temperature of neat EP was 93.0°C , which is a little lower compared with that of the Graphene/EP with a temperature value of 95.8°C . The increasing of glass transition temperature of modified sample can be attributed to the reason that graphene fillers surrounded by EP matrix provide an effective thermal dissipation path, while the efficient dissipation path depends on the cross-linking density.

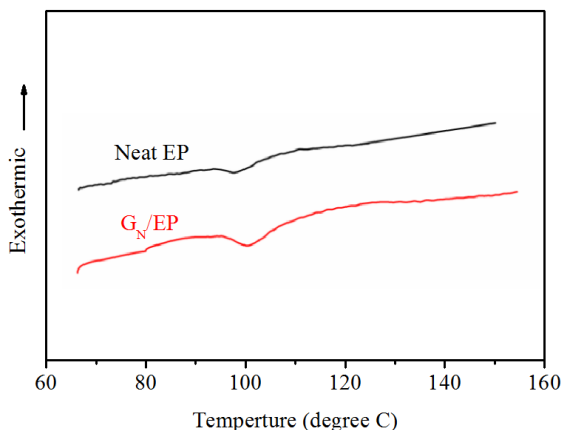


Figure 3. The DSC curve of samples

The measurement of thermal conductivity of modified sample shows a large improvement with its thermal conductivity of 0.267 W/mK which is about 157% times higher compared with that of the neat EP which has a thermal conductivity of 0.17 W/mK .

3.3 ELECTRIC PROPERTIES

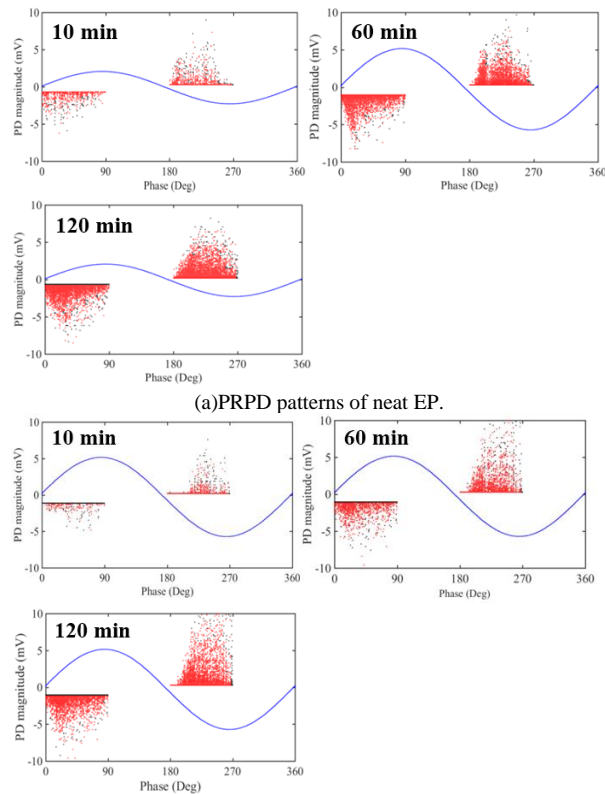
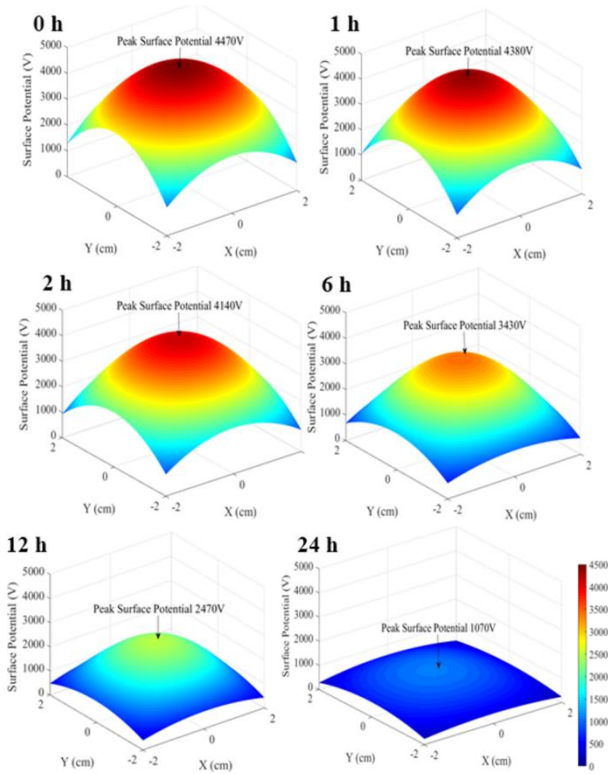


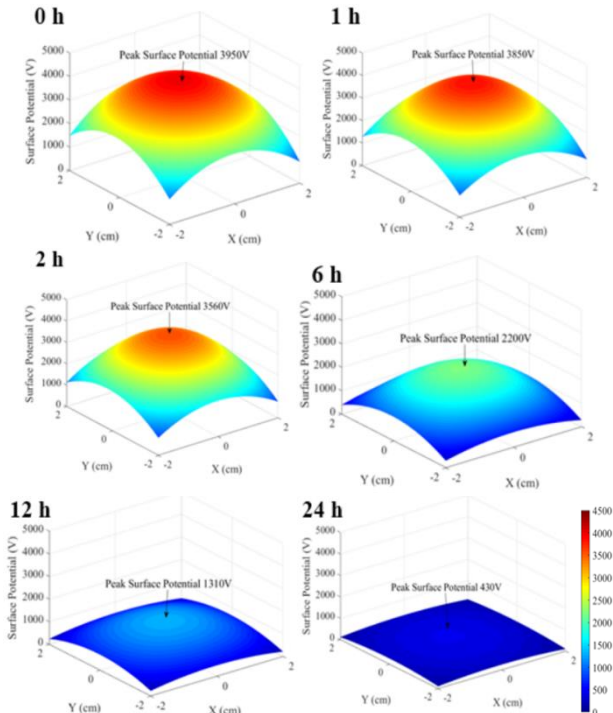
Figure 4. Patterns of phase resolved partial discharge (PRPD) for experimental samples.

During the PD measurement, the samples were tested under PD condition for 10 minutes, 30 minutes, 60 minutes and 120 minutes and the PRPD patterns were collected respectively. Figure 4 shows the phase resolved partial discharge (PRPD) patterns of the neat EP and the graphene doped EP. It is found that PD pulse signals in the phase diagrams were distributed between the range of 0° to 90° as well as 180° to 270° . Comparing the PRPD pattern of neat EP and graphene/EP, it is observed that the PD inception voltage and the density of PD pulses of graphene/EP are lower than that of the neat EP. However, not much difference can be found regarding the PD amplitude obtained by both samples.

The surface potential distribution and the peak value of surface potential for experimental samples can be found in Figure 5 and Figure 6. From Figure 5, it can be found that the surface potential distribution presents a bell-shaped distribution. Meanwhile, the surface potential decreases with the increase of time and researches less than 1 kV after 24 hours. From Figure 6 it can be found that the peak surface potential of graphene/EP is about 4 kV, which is smaller than that of the EP with a peak surface potential of more than 4.5 kV. The decreasing rate of the peak of the surface potential for both samples increases during the first few hours and then decreases after 15 hours.



(a) Surface potential distribution after different time duration for EP.



(b) Surface potential distribution after different time duration for G/EP.

Figure 5. The surface potential of (a) EP and (b) G/EP samples at 0h, 1h, 2h, 6h, 12h, 24h after corona discharge.

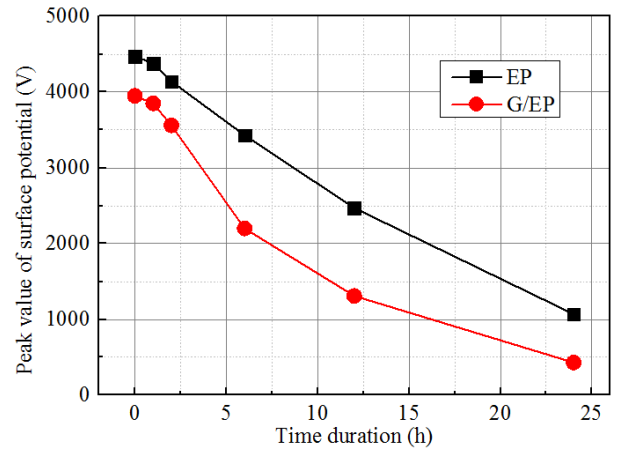


Figure 6. Peak value of surface potential of samples.

4 DISCUSSION

Thermal conduction in amorphous polymers is primarily based on phonons which are completed by the vibrations of a crystal lattice or lattice. Phonon scattering is prevalent in composites containing particles with smaller lateral dimensions as such composites have higher interface densities [21]. The intensity of phonon scattering is smaller, the thermal conductivity is higher; whereas the scattering intensity is higher, the thermal conductivity is smaller. The phonon scattering is caused by factors such as non-resonant vibration of the polymer molecules and the crystal lattice, defects in the interface of the polymer, and defects in structures. On the other hand, the thermal conductivity of the polymer material is also related to the number of polar groups and the degree of polarization of the polar group dipole moment. There are several possible reasons that account for the enhancement of thermal conductivity after the addition of graphene. Firstly, strong sp² covalent bonding of graphene leads to a lattice vibration. Therefore, the phonon scattering is just as small, which endows it with outstanding thermal conductivity. the formation of the dissipation pathways in the EP matrix due to the layer structure of graphene results in significant enhancement of the thermal conductivity. Meanwhile, low thermal boundary resistance between graphene and matrix can also be generated.

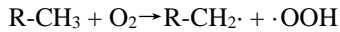
PD activity in the void of dielectrics is considered as one of the main causes of insulation breakdown and electrical degradation [22]. The initiating electron which is caused by natural high energy radiation quanta via either photo ionization in the gas or due to detrapping from the cavities interface. Then the electric field reaches PD inception field strength which is sufficient to induce an electron avalanche [22]. It is generally accepted that adding inorganic nanoparticles into polymer matrix is an effective method to improve PD resistance property of polymer insulation [23-24]. On the one hand, the high electric conductivity of inorganic nanoparticles are likely to promote charges dissipation and reduce the charge induced field, resulting in a rise of partial discharge inception voltage and a decrease of PD intensity. On the other hand, when nano particles are dispersed into the resin matrix, the resin acts as a scattering site which promotes the electron to transfer energy [25]. Adding the graphene promotes formation of conductive pathways which can quickly transfer charges. In addition, the

interface was formed by adding filler in which the charge was accumulated. Thus, the local electric field of charged sites would be decreased and the PD could be restrained. Therefore, higher voltage is required for PD to take place using graphene/EP sample.

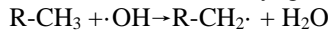
The PD process is not only determined by the gas in the gaps, but also by the surrounding material around the gaps. Meanwhile, the space charge produced by the PD strongly affects the activity of PD through changing the electric field in the cavity. The electric field due to the remaining space charges after the first PD is opposed to the applied field. Thus, it is impossible for another PD to occur in this half cycle, that is, the phases between 90°- 180° in the PRPD pattern[26]. When the polarity of the impulse voltage reverses, the electric field strength in the air gap changes to the superposition of the applied field and the field due to space charge accumulation. Therefore, the next PD occurs before the applied field reach the inception field strength.

For the surface of insulation, due to the combined action of humidity and the degradation products of air caused by discharge, there has an increase on the surface conductivity which can accelerate the diffusion of surface charges. The molecular chain would break when the energy of electrons and photons are higher than that of bond energy of material. Meanwhile, high chemical activity ions $\cdot\text{H}$ and $\cdot\text{O}$ can change the chemical structure and physic property during the discharge process. The degradation of epoxy resins caused by discharge in the atmosphere can be explained by the following reactions [28].

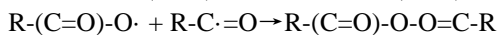
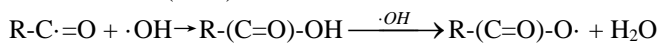
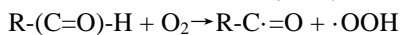
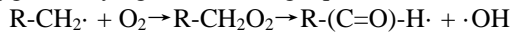
As shown in figure 7, firstly, activated oxygen attack on the methyl group:



or hydroxyl radical attack on the methyl group:



Then, the methylene radical on the resin chain reacts further with oxygen, obeying the following equations:



Further, the hydroxyl groups of branched chains are oxidized to carboxyl groups:

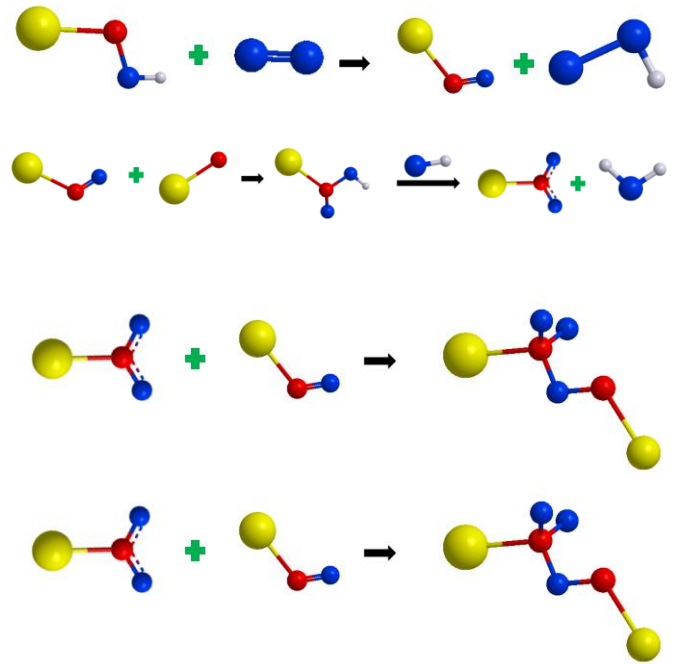
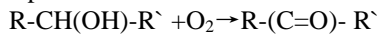


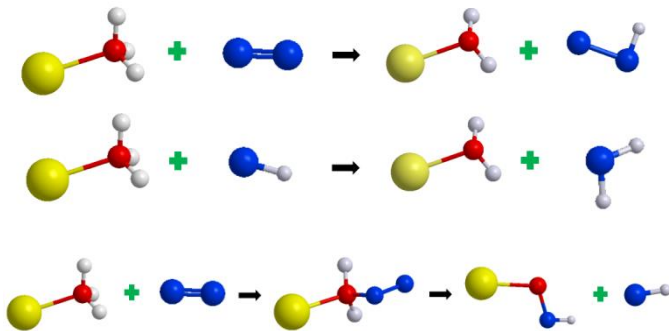
Figure 7 Schematic expression of chemical reaction process (Yellow, red, blue and white balls are R group, C, O and H atoms, respectively)

During the interaction with activated oxygen, hydroxyl and nitrous oxide species, EP can be aged more quickly. Meanwhile, the degradation can be more severe in humidity air than in dry air. According to the water contact angle measurement results, the graphene/EP sample has a better hydrophobicity which can effectively prevent water from absorption and permeation. In this case the oxygen permeability can also be restrained and the degradation of the materials can thereby be postponed.

According to the Wenzel-Cassie model, the contact angle is smaller for hydrophobic materials and the surface would be rougher for a more hydrophobic surface within a certain range of surface roughness [31]. A rougher surface can cause the serious charge accumulation due to a dense electric field created by local surface protrusions. Meanwhile, due to the excellent electric conductivity of graphene, the volume resistivity is decreased after doping with graphene. The lower volume resistivity of the material can prompt charge decay rate and restrain charge accumulation, which can prevent the creation of dense electric field.

5 CONCLUSION

Graphene fillers were doped into EP to modify the property of EP used in the encapsulation field for electric devices. The doping of graphene with a mass ratio of 0.1 wt% can increase the thermal conductivity of EP by 1.6 times. The increase in surface charge decay rate verifies an improved property in resistance of anti-static. Meanwhile, the PD activity of the sample after doping with graphene The findings in this paper provides a reference to develop a variety of outstanding thermal conductivity materials and approve the practicability of graphene/EP for using in the encapsulation layer of electronic devices. Further researches on the mechanical strength, fatigue rupture performance and



elongation at break (EAB) properties of graphene/EP composites is currently underway and will be reported in future, which is really important to the application of graphene composites in the field of encapsulation of electronic devices.

ACKNOWLEDGMENT

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