Influence of Octavinyl-Polyhedral Oligomeric Silsesquioxane on the Electric Treeing Resistance of Polypropylene

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***Abstract*—Electric treeing is the electrical pre-breakdown phenomenon which can degrade the insulating ability and limit the lifespan of solid insulations. In this paper, the research work investigates the influence of introducing OvPOSS (*Octavinyl-*Polyhedral oligomeric Silsesquioxane) nanoparticles on tuning the electric treeing characteristics of polypropylene (PP) under the pulse voltage with the frequency of 200 Hz, including the inception voltage for treeing growth, the electric treeing propagation rate under the pulse voltage of 30 kV. Some measurements, including scanning electron microscope (SEM) and thermal stimulated depolarized current (TSDC), were utilized to analyze the mechanism of the nanoparticle effects of OvPOSS. Finally, the experimental results showed that the addition of OvPOSS nanoparticles can obviously increase the inception voltage of treeing growth and suppress the treeing propagation under 30 kV pulse voltage due to the introduction of deep traps in the OvPOSS/PP nanocomposites.**

Keywords*—*electric treeing growth, polypropylene, octavinyl-POSS, pulse voltage, inception voltage.

I. INTRODUCTION

Depending on the advantages of low cost, good mechanical properties, excellent electrical performance, low dielectric loss and dielectric constant [1, 2], polypropylene (PP) has attracted considerable attraction for developing a new environmental-friendly insulation material. In the past two decades, many reports showed the electrical performance of polymers, including breakdown strength, dielectric loss and space charge characteristics, can be obviously enhanced by the addition of nanoparticles [3-7].

From the paper [8], the nano-silica can improve the electrical performance of LDPE but poor compatibility of silica-LDPE could cause the severe agglomeration which degraded the electrical performance of XLPE. In a different research, the molecular structure of POSS has a nano-silica cage in the nanometer scale with the bond of Si-O-Si and organic side group. The main cage of POSS can provide the similar role as traditional nano-silica and the organic function group can improve the nanofiller-polymer compatibility and adhesion. In this point, the nanodielectrics properties of various POSS, including octamethy-POSS (OmPOSS), octaisobutyl-POSS (OibPOSS), and Isobutyl-POSS (IoPOSS) in the polymers have been investigated as fundamental research [9] [10]. The results indicated introducing POSS into polymers can increase the breakdown strength and suppress the space charge accumulation under DC conditions.

In this paper, the inception voltage was measured to show the resistance to treeing initialization and the electric treeing length was pictured to estimate the propagation rate of treeing aging process. After that, the effect of OvPOSS on the electric treeing characteristics under the pulse voltage were investigated. It was found that the inception voltage of electric treeing inception increases by introducing 1.0 phr OvPOSS. The rate of treeing growth was significantly suppressed under the pulse voltage of 30 kV, 200 Hz and 30% duty circle, which was relevant to the deeper trap introduction.

II. MATERIAL PREPARATION AND EXPERIMENTS

A. *Materials*

The based polymer, isotactic polypropylene (iPP) with the density of 0.92 g/cm^3, was provided by SK Chemicals, South Korea. The OvPOSS nanoparticles were supported by Zhengzhou Alfache, Co., Ltd., China. The xylene with a purity of ≥99% was purchased from Aladdin Industrial Inc., China.

B. *Sample manufacture*

The powder of OvPOSS/PP NCs was manufactured by the xylene solution blending. This solution method depends on the situation that the xylene can dissolve OvPOSS and PP then the OvPOSS and PP can be mixed under the molecular level in xylene solution. After the production of NCs powder, the cube samples with inserted needle were produced by the compression moulding at the temperature of 200 °C and the pressure of 15 MPa and then cooled down to the room temperature under 10 MPa within 5 min. The radius of needle is 5 μm and the distance between needle and the ground was controlled to 2 mm ± 0.1 mm, shown in Figure 1.

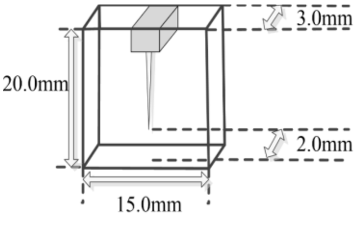

Fig. 1. Schematic of a treeing cube specimen with inserted needle

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C. *Experimental design*

The data of electric treeing growth were collected through the tree inception voltage measurement, the growing rate of electric treeing in the cube of OvPOSS/PP NCs. The scanning electron microscope (SEM) was used to observe the dispersion of OvPOSS nanoparticles in the bulk of NCs and the trapping characteristic would be characterized by the result of the thermal stimulated depolarized current measurement.

Firstly, the cube sample was immersed in the silicon oil in order to avoid the flashover during the test. Then the electric treeing test was carried on under 30 °C. Thirdly, the power source shown in Fig.2 contained a signal generator and HV amplifier. The signal generator produced a 6.0 V pulse signal with the frequency of 200 Hz and the duty circle of pulse signal is 30%. Then, the pulse would be amplified to 30 kV by the HV amplifier and applied to the cube sample to simulate the operation overvoltage in the real HVDC application. Under the applied pulse voltage, the electric treeing growth will be observed by a digital microscope and shown in the computer.

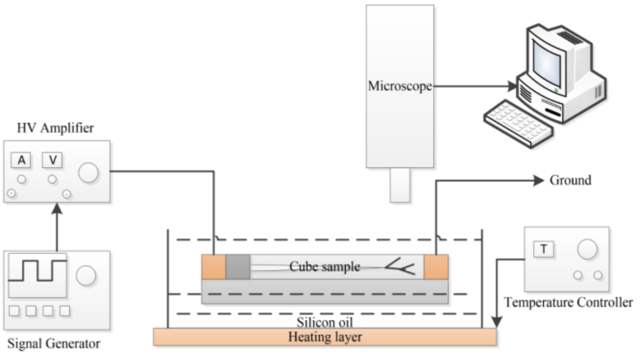

Fig. 2. The design of experimental platform for electric treeing detection

III. EXPERIMENTAL RESULTS AND ANALYSIS

A. *SEM characteristics of OvPOSS/PP NCs samples*

The SEM image of OvPOSS/PP NCs is captured by Hitachi 8010,Japan and shown in Fig.2. The small agglomerates have been marked. It can be found that there is no significant agglomerates occurring and the diameter of most clusters is below 300 nm in the NCs. The image indicates the homogeneous dispersion of OvPOSS have been achieved successfully and the adverse effect of severe agglomerates with the diameter of over 1.0 μm has been avoided.

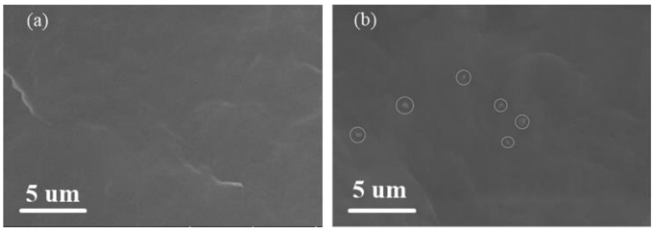

Fig. 3. The SEM images of (a) pure PP and (b) OvPOSS/PP NCs with 1.0 phr

B. *Trap level distribution of OvPOSS/PP samples*

The film samples are placed in the broadband dielectric spectrometer (Novocontrol GrmbH Concept 40, Germany) and the surface of film samples was covered by a gold film. Firstly, the sample was polarized under 50 kV/mm at 50 °C for 40 min, then cool down to -100°C by -10 °C/min. Next, the samples were depolarized by 5 min so that the surface charges can be removed. Finally, the thermal stimulated current was measured by the galvanometer with the temperature increasing to 100 °C.

The TSDC result is shown in Fig. 4. Therefore, the analysis of TSDC result in the study mainly focuses on the electron trapping characteristics. Through the method from [11], the TSDC result can be transformed to give a relationship between trapping level and trapping density. In pure PP, there are two peaks of TSC, namely 0.94 pA at -10℃ and 0.69 pA at 58 ℃, which is corresponding to 0.75 and 0.91 eV while the two peaks of TSC in PP/OvPOSS NCs are corresponding to 0.75 and 1.02 eV. Because the first peak is related to the polymeric segmental relaxation by the glass transition, the trapping characteristics can be described by the second peak. It revealed that the addition of OvPOSS nanoparticles can introduce deep traps into the base PP. It results in the trapping level of the traps in 1.0 phr OvPOSS/PP NCs being much higher than pure PP and it indicates that more charges can be trapped in NCs samples, normally trapped by the OVPOSS nanoparticles. The mechanism of deep traps is relevant to the chemical bond of C=C in the side group of OvPOSS due to the sp2 hybrid orbitals. The strong negativities of oxygen atom in OvPOSS can attract the electron from Si atom in the cage and C atom through the induction effect. Also, OvPOSS can act as nano-SiO_2 in PP, which can introduce the deep traps by the interfacial effect between OvPOSS and PP [8, 12]. After the trapping procedure of charges, the trapped charges barriers can suppress the charge injection from the electrodes. In the samples, the charges are mainly electron not ions, because the size of electrons is much smaller than ions and the ions transport is much more difficult than electrons . This situation can be seen in many results of space charge measurements [9].

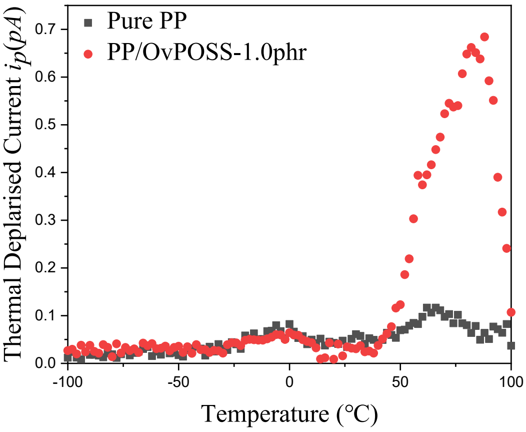

Fig. 4. The trapping level distribution transformed from TSDC measurements in OvPOSS/PP NCs

C. *Inception voltage and growth of electric treeing in OvPOSS/PP under pulse voltage*

The inception pulse voltage of electric treeing growth is defined for a voltage with 200 Hz and 30% duty circle which can start the electric treeing within 10 min and the voltage increases by 0.5 kV. The Weibull probabilities of inception voltage of electric treeing growth in PP and OvPOSS/PP NCs at 30°C can be calculated by the formula below [13].

$$P = 1 - \exp\left[-\left(\frac{V}{V_0}\right)^{\beta}\right] \qquad (1)$$

where:

P is the cumulative probability of tree inception

V is the applied pulse voltage with 200 Hz, 30% duty circle.

V_o is the critical value of inception voltage in Weibull distribution.

The critical value of inception voltage V_0 at the cumulative probability of 63.2% and shaping factor β are demonstrated in Tab. 1. The comparation of PP and OvPOSS/PP NCs reveals that the addition of OvPOSS nanoparticles increase the inception voltage from 19.8 kV to 21.9 kV.

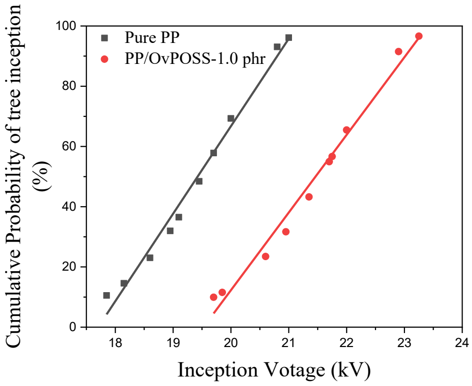

Fig. 5. The Weibull distribution of inception voltage of electric treeing growth

TABLE I. THE INCEPTION PULSE VOLTAGE OF ELECTRIC TREEING OF PP AND ITS NCS

Items	**Parameters of Weibull Distribution**	
	Critical Value (V_0)	***Shaping factor (β)***
Pure PP	19.8 kV	20.76
PP/OvPOSS	21.9 kV	21.00

For the long-term electrical insulation, the extension of electric treeing could decrease the lifespan of insulators. Normally, the electric treeing can be divided into branch type and bush type and the shape of electric treeing would be affected by the nanofillers dispersion, electric field and the distribution of crystalline and amorphous regions. The electric treeing propagation rate in the branch type is faster than the bush type [14]. Therefore, the effect of introducing OvPOSS can be demonstrated in the length and the shape of electric treeing.

For the comparation of electric treeing characteristics, the electric treeing image in 1 min, 5 min, 10min and 15 min are selected due to the fast growth of electric treeing in the initial step. As Fig. 6 shows, the length of electric treeing increases fast as a branch type from the tip to the ground within 1 min, then changes to the branch-bush types from 5 min to 25 min. After the addition of 1.0 phr OvPOSS, the bush type of electric treeing prevails in PP/OvPOSS NCs and the growth of electric treeing in NCs is obviously slower than the pure PP.

Fig.7 shows the mechanism of how introducing OvPOSS can suppress the electric treeing. When the pulse voltage is applied, a large number of electrons would be injected quickly into the cube sample from the ground. Some excited electrons could trap and detrap in the shallow traps until they are captured by the deep traps. From the TSDC result, the addition of OvPOSS can introduce deeper traps with high trapping density into the sample. Therefore, these electrons could be captured by OvPOSS, then those trapped electrons could have the barrier effect on the electron injection from the ground and extraction around the needle. Hence, the mobility of electron could be greatly suppressed, then the electric treeing propagation would be reduced.

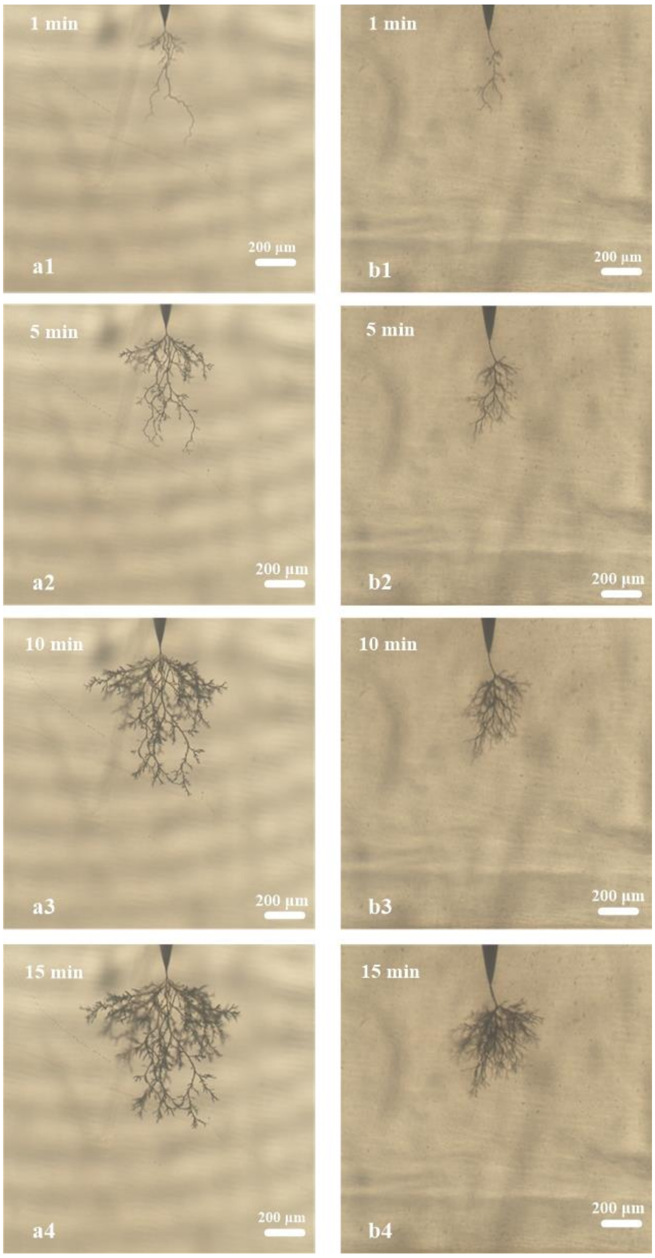

Fig. 6. Images of electric treeing growth of (a) pure PP and (b) PP/OvPOSS NCs in 1 min, 5 min, 10 min, 15 min

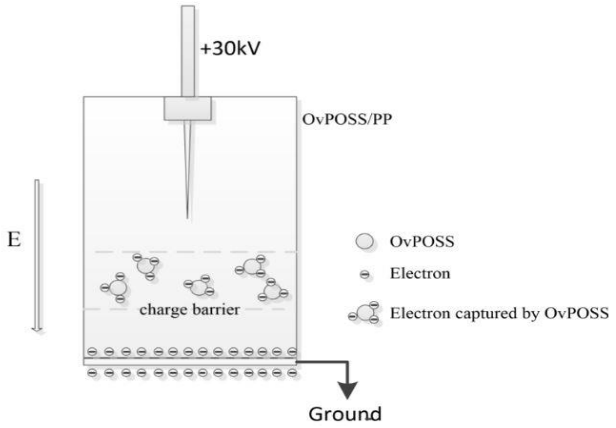

Fig. 7. The analysis of charge barrier effect

IV. CONCLUSION

The addition of OvPOSS nanoparticles is aimed to impede electric treeing in the base PP insulation material. The improvements to treeing characteristics have been investigated, including the trapping level enhancement, the increase of treeing inception voltage and the lower propagation rate of electric treeing. The mechanism for treeing suppression is related to the charge barrier built by the captured electrons. These charge barrier can suppress the electric treeing propagation by reducing charge mobility.

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