Charge trapped mechanism for semi-crystalline polymer electrets: quasi-dipole model

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Abstract: Polymer electrets are increasingly getting application in a very wide range. However, its charge trapped mechanism is still poorly understood. It is always challenging how to improve its charge trapped ability and to enhance its performance stability. For example, melt-blown polypropylene (MBPP) electret fabric is highly concerned and has become the leading material for air filtration because of its remarkable ability to capture the particulate with aerodynamic equivalent diameter ≤ 2.5 μm (PM2.5) [15–17]. Its outstanding filtration performance mainly depends on the electrostatic field established by the charges-trapped within MBPP fabric [18, 19]. However, the charge decay easily takes place. It is very urgent to clarify the charge trapped phenomena from microstructure for improving the performance of electret materials.

Several studies have reported that reason of the charge decay is ascribed to the chemical reaction of solvents with polypropylene (PP) surface [20], conductance or ion masking on PP surface [21, 22]. The charge trapped performance has been expected to have a connection with its crystalline features such as crystalline form [23, 24], the amorphous-to-crystalline ratio [25, 26], crystallite grain size [27] and spherulites features [28, 29] in a semi-crystalline polymer material such as PP. However, these works just stay on the description of the charge trapped phenomena and do not involve in the charge trapped mechanism. Although the energetic band model is a good theory to explain the charge behaviour in conductors and semiconductors [30], it is not reasonable for the polymer because of the particularity of polymer structure. The charge trapped mechanism in polymer electrets is still poorly understood.

Besides, the charge trapped mechanism for the semi-crystalline polymer is also paid close attention by the researcher of high-voltage electric technology because it is implicated in the dielectric breakdown, lifetimes of cables and the better design of electrical installations [31]. In the proposed charge trapped mechanism, the ‘defect’ is the main concept and leads to the accumulation of charge in cables. It has been suggested that both physical and chemical defects are the main charge traps [32]. The charge trap is divided into shallow and deep energy levels [33]. Shallow energy level arises from physical or conformational defects in the amorphous polymer. And the deep energy level is associated with chemical defects or impurities. However, the ‘defect’ is an abstract concept and very difficult to be observed by means of an experiment despite having been correlated to the chemical composition and structure of the polymer.

In our previous paper, it was proposed that the charge trap source was mainly ascribed to the charged crystallite grain in MBPP electret fabrics [34], which was proved by the experiment results that the trap concentration increased with rising crystallinity and fineness of crystallite grain. In this paper, the charged crystallite grain is viewed as the quasi-dipole through considering of the crystalline structure difference among different crystallites for the semi-crystalline polymer electrets. Its physical principle is based on the interfacial polarisation between crystallite and amorphous region due to their different conductivity. The melt-blown PP (MBPP) electret fabrics with α or mesomorphic crystallite are used as a model material to verify the rationality of the quasi-dipole model. Their thermally stimulating discharge (TSD) current and X-ray diffraction (XRD) spectra are measured. The improvement of charge-trapped performance is ascribed to the crystalline structure transformation from mesomorphic crystallite to α crystallite in MBPP electret fabrics.

2 Charge trapped mechanism

2.1 Semi-crystalline structure feature of polypropylene

Polypropylene, as a typical semi-crystalline polymer, is composed of crystallite and amorphous region introduced by twisting and branching. It was known that the crystallite of PP may be polymorphic phase such as α-monoclinic, β-hexagonal, γ-orthorhombic and mesomorphic phase depending on the crystallising condition [35, 36]. It has been suggested that the α-phase of PP exists in a mono-clinic lattice with alternate layers of left- or right-handed helices [37]. The α-phase is stable thermodynamically and has good mechanical strength, resulted from the cross-hatched lamellar morphology and compact stacking of molecular chains. The mesomorphic crystallite of PP has the degree of ordering with the intermediate state between the crystalline phase and the amorphous phase, which is characterised...
regularity. The electrical conductivity in the RAR is indeterminacy because of the interface between CR and MAR, namely the rigid amorphous region (RAR). In the RAR, the conformation regularity of segments neighbouring to the CR is higher than those close to the MAR. This is a gradual change region of segment conformation regularity. The electrical conductivity in the RAR is indeterminacy because of the continuous variation of conformation regularity. Namely, the variable electrical conductivity of the RAR is responsible for the difference of charge-trapping performance in MBPP fabrics.

2.2 Quasi-dipole model

When PP is charged under a direct current field, the charges tend to pile up on the interface between crystallite and amorphous region because the interface polarization takes place, where the normal drift of charges is hindered because the electrical conductivity is relatively small for crystallite region and relatively large for the amorphous region [34]. As a result, every grain of crystallite becomes a dipole. These quasi-dipoles become the main charge trapped source. The energy level of charge trap has a dependence on the interfacial conductivity between crystallite and amorphous region. And charge-trapped stability is correlated to the regularity of the crystalline structure. Based on this viewpoint a quasi-dipole model for a semi-crystalline polymer is proposed as shown in Fig. 1. At the top is the quasi-dipole schematic diagram of charge piled up at spherulite surface, and the bottom part is the interface change of the spherulite area.

According to the three region model of crystallite, RAR is a gradual change region of segment conformation regularity. The electrical conductivity in the RAR is indeterminate because of the continuous variation of conformation regularity. Namely, the variable electrical conductivity of the RAR is responsible for the difference of charge trapped performance. In the mesomorphic crystallite, one of the structural features is that a large amount of RAR coexists with relatively little MAR, the content ratio between RAR and MAR is about 2. Therefore, the electrical conductivity border between the CR and the amorphous region is vague due to the thick RAR. The quasi-dipoles originating from charged crystallite grain are unsecured. The charge trapped ability is relatively poor. In the α-crystallite, the RAR is very thin. The electrical conductivity border between the CR and the amorphous region is relatively clear. Its crystalline grain becomes an integrated quasi-dipole under the action of an external electric field. The charge trapped ability is relatively good. It is concluded that the more regular the crystallite grain structure the better charge-trapped stability is. Therefore, the PP electrolyte materials with α-crystallite display better charge-trapped stability.

3 Experiment results

3.1 Control of crystalline structure in MBPP fabrics

The mesomorphic crystallite of PP is generally obtained by rapid quenching the melt at low temperatures, or, more precisely, by cooling the melt at cooling rates higher than 100 Ks⁻¹, generally between 100 and 1000 Ks⁻¹[41]. It has been found in our previous studies that the MBPP fabric made in the custom-designed micro melt-blown machine mainly is the mesomorphic crystallite because of very fast cooling rate (about 500 Ks⁻¹) and the insufficient shearing force [42]. By means of an annealing process, the mesomorphic crystallite may transform to α-crystallite [43]. The proportion of α and mesomorphic crystallite in MBPP fabrics may be modulated through an annealing process at elevated temperature. In this research, the MBPP fabric is annealed at the temperature of 60, 90 and 120°C for 10, 20 and 30 min, respectively. The wide-angle XRD (WAXD) measurement is performed to detect the crystal structural transition.

Fig. 2a is the Wide-Angle X-ray Diffraction (WXRD) spectra of the MBPP fabrics annealed at 60°C. It is shown that there are two strong wide peaks, wherein 2θ values are 15.0° and 21.3°, respectively, which are the characteristic peaks of mesomorphic crystallite [44]. No obvious change of WXRD spectra is observed when the samples are annealed for more time, although the sketch of other peaks appears due to a glass transition-like behaviour resulting from the chain reorganisation of the interfacial region of the mesophase [45]. This shows the main constituent of crystallite in MBPP fabrics is still mesomorphic phase as same as no annealed sample.

When annealing temperature rises to 90°C, the transformation of the crystalline structure becomes obvious as shown in Fig. 2b. Four strong wide peaks gradually appear, and enhance with the extension of annealing time from 10 to 30 min. Their 2θ values are 14.3°, 17.2°, 18.7° and 21.8°, corresponding to the lattice planes (110), (040), (130) and (041), respectively, which are the characteristic diffraction peaks of α-crystallite with monoclinic configuration for PP [46, 47]. The relative intensity for lattice plane (111) is significantly reduced and its diffraction peak overlaps with that of (041). Therefore, it can be pointed out that annealing at 90°C leads to transformation from mesomorphic-crystallite to α-crystallite in MBPP fabrics.
When annealing temperature rises to 120°C, four strong wide peaks appear in the XRD spectrum of the MBPP fabric as shown in Fig. 2c. Their 2θ values are 14.3°, 17.2°, 18.7° and 21.8°, respectively, which are the characteristic peaks of α-crystallite. The change of WXRD spectrum is not obvious when annealing time is extended from 10 to 30 min. This result shows that the proportion of α-crystallite has almost reached its maximum after annealed at 120°C for 10 min.

Based on the above results, it can be known that the crystallite of the MBPP fabrics mainly keeps mesomorphic phase when annealed at 60°C, and quickly transform from mesomorphic phase to α crystaline phase when annealed at 120°C. For the MBPP fabrics annealed at 90°C the proportion of α and mesomorphic crystallite is a function of annealing time, in which α-crystallite gradually increases and mesomorphic-crystallite gradually decreases with the extension of annealing time.

The mesomorphic – α crystallite transformation process can be observed by polarised optical microscopy (POM). Fig. 3 shows that optical micrographs of the crystallite transformation process during the annealing at 90°C. The mesomorphic crystallite usually has a very faintish birefringence because of irregular crystalline structure, while α crystallite have relatively bright birefringence because of the relatively regular crystalline structure due to very thin RAR layer. From photo A to photo D, the transformation of the crystalline structure is very obvious. The birefringence is faintish and crystallite is loose in the without annealed sample with mesomorphic crystallite as shown in Figs. 3a and b. After the sample is annealed for 20–30 min as shown in Figs. 3c and d, mesomorphic crystallite transform α crystallite, the birefringence become bright and the regular of crystallite is improved. This result is consistent with the conclusion of WAXD spectra shown in Fig. 2b.

### 3.2 Analysis of TSDC spectra

TSDC has been widely used to the investigation of charge trapped performance in electret materials according to the peak temperature of TSDC spectra [48]. The higher the TSDC peak temperature, the better the electret charge-trapping performance is [49]. The TSDC spectra of the samples after annealed at 60°C for 10, 20 and 30 min are shown in Fig. 4a. The peak temperature of TSDC spectra is around 86°C, which is the temperature for samples to release charge. Fig. 4a also shows the change of peak temperature is little when the annealing time is extended, which shows the charge-trapping performance of MBPP electret fabrics is not improved too much during annealing at 60°C.

Compared with the results in Fig. 2a, in which the proportion of mesomorphic-crystallite does not increase when the annealing time is extended, it can be argued that the special discharge peak temperature of MBPP electret fabrics with mesomorphic-crystallite is about 85°C. As a comparison, Fig. 4a also displays the TSDC spectrum of the uncharged sample, in which no current peak is observed. This implies that uncharged sample does not have the characteristic of electrets.

The TSDC spectra of the samples annealed at 90°C for 10, 20 and 30 min are shown in Fig. 4b. The peak temperature of TSDC spectra moves to higher temperature with the extension of annealing time. When the annealing time is 10 min, the peak temperature of the TSDC spectrum is about 94.9°C. When annealing time prolongs to 20 and 30 min, the peak temperature increases to 113.0 and 129.5°C. This result indicates that the charge trapping capacity of MBPP electret fabrics is gradually improved during annealing. Meanwhile, the proportion of α-crystallite increases when annealing time is extended from 10 to 30 min as shown in Fig. 2b. Therefore, it can be concluded that there is a significant correlation between electret charge trapping performance and crystallite feature. The larger the proportion of α-crystallite, the higher the TSDC peak temperature is. Better electret charge trapping performance can be ascribed to the transformation from mesomorphic-crystallite to α-crystallite. The charge trapping stability of α-crystallite is better than that of mesomorphic-crystallite.

The TSDC spectra of the samples after annealed under 120°C for 10, 20 and 30 min are shown in Fig. 4c. It shows that the change of the peak temperature in TSDC spectra is a little, which increases from 120.2 to 125.8°C when annealing time is extended from 10 to 30 min. The results show that the improvement of charge trapped capacity has been almost finished at the initial 10 min. Compared with the results in Fig. 2c, it can be argued that the special discharge peak temperature of α-crystallite MBPP electret fabrics is about 123°C.
4 Discussion

4.1 Correlation between crystalline structure and TSDC spectra

To explain more clearly the correlation between the charge-trapping ability and crystalline features, the XRD and TSDC spectra of MBPP electret fabrics annealed for 10 min at elevated temperature are shown in Fig. 5. The component analysis on the quantitative crystallization performance according to XRD spectrum in Fig. 5a is carried out, assuming each spectrum consists of three components: α-crystallite, mesomorphic-crystallite and amorphous. Crystallinity and grain size are calculated according to the method reported in [45]. The calculating results are shown in Table 1. Following results can be gotten:

(i) The size of crystalline grain increases with rising annealing temperature, which is only 2.89 nm for no annealed sample and increases to 5.6 nm for the sample annealed at 120°C. It is correlated to the proportion increase of α crystallite, which is from 35.8 to 89.7%. For no annealed sample, the main crystallite is mesomorphic, which crystal border is loose and vague. After the sample is annealed at elevated temperature, mesomorphic crystallite gradually transformed α crystallite. The regularity of crystallite is improved. The size of the crystalline grain increased.

(ii) Compared with the non-annealing sample, the α-crystallite proportion in the sample annealed at 120°C rise 33.9% and crystallinity increases only 4.8%. This hints that the annealing process mainly leads to the transformation from the mesomorphic phase to α-phase (meso-α transformation). This result also proves that the improvement of charge-trapped stability is correlated to the meso-α transformation. And the crystallinity is correlated to the trap concentration according to our previous research [43]. This also shows every crystallite grain is equivalent to a charged dipole. The higher the structural regularity of the dipoles, the better the charge-trapped stability is.

(iii) The TSDC peak temperature and α crystallite proportion are both the function of annealing temperature. When annealing temperature rises from 60 to 120°C, TSDC peak temperature and α crystallite proportion both increase a lot. For no annealed sample, TSDC peak temperature is the lowest and α crystallite proportion is the least. It has pointed out earlier that the higher the TSDC peak temperature, the better the electret charge trapping stability is. Therefore, these results indicate that the charge-trapping stability of MBPP electret fabrics is gradually improved with increasing α crystallite proportion. It is also hinted that the charge trapping stability of α crystallite is better than mesomorphic crystallite. These results are consistent with the conclusion from the quasi-dipole model. The α-crystallites with the more perfect structure in MBPP electret fabrics leads to the better charge-trapped stability.

4.2 Consistency of experiment results with analysis from quasi-dipole model

From the above experiment results, it can be concluded that the effect of annealing on improving the charge-trapped stability in PP is ascribed to the increase of α-crystallite proportion. The reason can be correlated to the quasi-dipole model.

Have pointed out above, the local correlation between chains inside each small aggregate in mesomorphic crystallite is similar to the crystal structure characteristic of the monoclinic α-phase in PP. Mesomorphic-crystallite acts as precursors of subsequent crystallisation of α-crystallite. The transformation from mesomorphic crystallite to α-crystallite is in two steps: first, the annealing below 80°C leads to achieving better registration of helices chain; then, the annealing in the temperatures of range 80–150°C, the helices arrange in the lateral direction [38]. The mesomorphic-crystallite begins to transform into α-crystallite. In this process, the segments which have correct chirality but incorrect position adjust their position by moving (translation) and the chains with the correct position but incorrect chirality adjust their chirality by helical rewinding. This is responsible for the
decrease of RAR content because partial RAR segments neighbouring to the MAR experience glass transition and transfer to MAR. The RAR segments with higher conformation regularity which are close to the mesomorphic region are reserved. This leads to the improvement of the conformation regularity PP crystallite. And the electrical conductivity border between the CR and amorphous region gradually becomes distinct. The quasi-dipoles become integrated and the charge trapped performance is improved.

5 Experiment methods

5.1 Preparation of MBPP fabric with mesomorphic-crystallite

The MBPP fabric with mesomorphic-crystallite is made from granule isotactic PP (with melting index of 1500 from Shandong Dawn Polymer Co. Ltd of China) by custom-designed micro melt-blown machine as shown in Fig. 6. The processing conditions are adjusted by a computer. Unless especially pointed out, the fabric receiving distance is 16 cm, PP melt temperature and pressure are βγ0°C and 0.γ MPa, respectively, hot air temperature and pressure are ββ0°C and 0.β MPa, respectively. The resulted MBPP fabrics have an average face density of β5 g/m², the average thickness of 0.β6 mm and the average fibre diameter of 5.07 μm.

5.2 Formation of electrets

The MBPP electret fabric is formed via a corona charging device with an adjustable electric field [50]. The charging conditions of the electric field (Ep) of −γ0 kV and charging time of 1 min are taken at room temperature.

5.3 Modulation of crystalline structure

The proportion of α- and mesomorphic crystallite in MBPP fabric may be modulated through an isothermal annealing experiment. The MBPP fabric is put to a thermostat and annealed for 10, 20 and 30 min at the temperature of 60, 90 and 120°C, respectively, then taken out and stored in a drying cabinet with a temperature of β5°C and relative humidity of 45%.
The charge trapped performance in semi-crystalline polymer electrets such as PP may be explained by the quasi-dipole model, which correlates to the increase of α-crystallite proportion. The charge-trapped stability in MBPP fabrics with α-crystallite is better than one with mesomorphic-crystallite.

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8 References
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