

Investigations on the Interactions between Li-TFSI and Glass Fibers in the Ternary PP/GF/Li-TFSI Composites

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Abstract The polypropylene/glass fiber (PP/GF) composites with excellent antistatic performance and improved mechanical properties have been reported by incorporation of a very small amount of the organic salt, lithium bis(trifluoromethanesulfonyl)imide (Li-TFSI), into the PP/GF composites. It was considered that GF could play the role as the pathways for the movements of ions in the ternary composites. In this work, the interactions between Li-TFSI and glass fiber and the effects of such interactions on the physical properties of the composites have been systematically investigated. Three types of glass fibers with different —OH group concentrations have been prepared in order to compare the interactions between GF and Li-TFSI. It was found that the —OH group concentrations on the surface of glass fiber have significant effects on interactions between glass fibers and Li-TFSI. Such interactions are crucial for both the antistatic and mechanical performances of the final PP/GF/Li-TFSI composites. The investigation indicated that the GF with high —OH group concentrations confined the movement of TFSI[−], which decreased the antistatic properties of PP/GF/Li-TFSI composites. On the other hand, the GF with low —OH group concentrations inhibited the absorption of Li-TFSI onto the GF, which also hindered the formation of Li-TFSI conductive pathway. The best antistatic performance of the ternary composites can be achieved at the intermediate —OH concentrations on the GF.

Keywords Polypropylene; Glass fiber; Li-TFSI; Antistatic performance

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INTRODUCTION

The antistatic modification of polyolefin has attracted significant attentions for many years due to the wide industrial applications^[1–3]. PP has very low moisture absorption and shows high electric insulation, and hence, it is easily charged by static electricity^[4]. Considerable investigations have been undertaken on the antistatic PP materials by the incorporation of antistatic agents^[5–11] and conductive fillers^[3, 8, 12–15]. Conductive fillers, such as carbons, carbon fiber and conductive mica, can dissipate the accumulated charges by the conductive pathway or network in the polymer matrix, but their applications are limited by the drawbacks of high percolation threshold, dark color and poor processability^[3, 8, 12–15]. The amphiphilic molecules as the internal antistatic agents have many advantages over the conductive fillers because they usually enhance the processability and the mould releasing ability except for the improved electric conductivity. Nevertheless, the antistatic effects of these additives are not permanent and strongly moisture dependent^[5–11]. Organic salts (OS) (some are the room temperature ionic liquids (RTILs)) exhibit low toxicity, relatively low melting points, negligible vapor pressure, high

thermal and chemical stabilities, high ionic conductivity, and a broad electro-chemical potential window^[16–21]. They have also been used as the antistatic agents for polymers. However, the OS are usually not miscible with polymers due to the greater difference in the polarity between polyolefin and OS. Therefore, significant antistatic performance can only be achieved by the high loadings, which will inevitably induce the bleeding of the OS from the polymer matrix.

Very recently, we have found that glass fibers improve the antistatic properties and enhance the antistatic stability of lithium bis(trifluoromethanesulfonyl)imide (Li-TFSI) incorporated PP^[22]. The preliminary investigations indicated that Li-TFSI had specific interactions with GF and was absorbed exclusively on the surface of GF. GF network in the PP matrix provided perfect pathways for the movements of ions, therefore the excellent antistatic performance was achieved for the PP/GF/Li-TFSI composites at the Li-TFSI loading of as low as 0.25 wt%, while the binary antistatic PP/Li-TFSI materials could only be achieved at the Li-TFSI loadings of more than 3 wt% and large-size aggregates of Li-TFSI were observed in PP matrix. Obviously, the excellent antistatic properties of the ternary PP/GF/Li-TFSI composites should be attributed to the synergistic effects of glass fiber and Li-TFSI. However, the specific interactions between GF and Li-TFSI and the effects of such interaction on the final antistatic and mechanical performance of PP/GF composites

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are still far from clear. We consider that the interactions between GF and the Li-TFSI are originated from the hydrogen-bonding interaction between —OH groups of GF and the ions of Li-TFSI. In order to elucidate the mechanism for the synergistic effects of GF and Li-TFSI, therefore, in this work we prepared three types of glass fibers with different —OH group concentrations. We investigate the interactions between the three types of GF and Li-TFSI followed by the characterization of the antistatic performance of the final PP/GF/OSs composites. We demonstrate that the surface characteristics of GF play the critical role for the properties of final PP/GF/Li-TFSI ternary composites.

EXPERIMENTAL

Materials

PP used was commercially available from Sumitomo Co. Ltd (Japan) with the trade name of AH561. Lithium bis(trifluoromethanesulfonyl)imide (Li-TFSI) was supplied by Solvay Co. Ltd (Belgium). The short glass fibers were kindly provided by Jushi Co. Ltd (China) with a type of 568H. Silane coupling agent (trimethoxy(propyl)silane) was purchased from Tokyo chemical industry Co. Ltd (Japan). All the samples were used as received.

Surface Treatment of Glass Fiber

The short glass fibers provided by Jushi Co. Ltd have the medium —OH concentration and were termed as GF-M- OH . The fibers were ignited at 450 °C for 4 h in a muffle furnace followed by sonication in ethanol for 5 min. After that, the ignited glass fibers were dipped into 10 wt% hydrochloric acid for 3 h. The obtained glass fibers have high —OH concentrations and were termed as GF-H- OH . To prepare the glass fibers with low OH concentration, the GF-M- OH was further coated by silane coupling agents. The sizing solution of coupling agent, ethanol and de-ionized water were taken in a mass ratio of 10:82:8. The solution was stirred for 1 h at room temperature with pH adjusted to 3.5 using acetic acid. After that, the GF-M- OH was dipped in the coating solution for 1 h. The glass fibers were then air-dried at room temperature for 5 h, followed by vacuum oven drying over-night at 110 °C. The obtained GF have low —OH concentration and were termed as GF-L- OH . All the GF were stored in a desiccator prior to use. For infrared examination, three types of GFs were dipped into aqueous solution of the Li-TFSI (a mixture of Li-TFSI and de-ionized water at a mass ratio of 1:5). The Li-TFSI absorbed glass fibers were then dried at 50 °C in vacuum oven over night before the infrared examination.

Preparation of PP/Li-TFSI and PP/GF/Li-TFSI Composites

All the components were kept dried under vacuum at 80 °C for 12 h before processing. All the composites were prepared by direct mixing of PP, GF, Li-TFSI in a batch mixer (Haake PolyLab QC), at 50 $\text{r}\cdot\text{min}^{-1}$ and 190 °C for 5 min. After melt-mixing, parts of samples were hot-pressed at 200 °C and 10 MPa into 500- μm -thick films, followed by the cold-pressed at room temperature. The obtained sheets were characterized directly. Other parts of samples were injected to a standard specimen of ISO 527-2-5A using a Haake MiniJet Pison Injection Molding System (Haake Germany) at 230 °C

with an injection pressure of 50 MPa. The mold temperature was 100 °C. The sheets were used for electrical conductivity measurements and injection molded specimens were used for the tensile tests.

Characterizations

The Fourier transform infrared spectroscopy (FTIR) measurements were carried out in transmittance mode on grinding samples by FTIR spectrophotometer (FTIR, Bruker Tensor). The FTIR spectra were recorded at a resolution of 2 cm^{-1} , and 64 scans from 4000 cm^{-1} to 400 cm^{-1} were averaged.

X-ray photoelectron spectroscopy (XPS) analysis was carried out using a Kratos Axis Ultra instrument with monochromatic Al $K\alpha$ X-ray source. Wide scan was investigated in the range of 1300–0 eV and the narrow scans were measured for the O1s, C1s and Si2p regions.

Thermogravimetric analysis (TGA, TA-Q500) was carried out at a heating rate of 10 $\text{K}\cdot\text{min}^{-1}$ from room temperature to 650 °C, in a high purity N_2 atmosphere.

The microstructure of cross-fractured surface of samples was observed using field emission scanning electron microscopy (FESEM, SEM-JSM 6700). An acceleration voltage of 3 kV was used for the samples and the fractured surface was coated with a thin layer of gold before the SEM observation.

Electrical conductivity was measured by an ultrahigh resistivity meter, with a piece of URS probe electrode (Model MCP-HT450) at 100 V. The sample thickness was about 500 μm .

Tensile tests were carried out using an Instron universal material testing system (Model 5966) at 23 °C, with a gauge length of 20 mm and crosshead speed of 5 $\text{mm}\cdot\text{min}^{-1}$.

A polarized optical microscope (POM, Olympus BX51-P) with a hot stage unit was used to study the morphologies of PP/GF composites. All the samples were heated to 200 °C.

RESULTS AND DISCUSSION

Characterization of Three Types of GFs

Figure 1 shows the FTIR spectra of silane coupling agent, GF-L- OH , GF-M- OH and GF-H- OH . The intense band at 867 cm^{-1} , which is assigned to the asymmetric bending vibration of $\text{Si—OH}^{[23]}$, was observed in all the three types of GFs, confirming the existence of Si—OH on the surface of glass fibers. Two new peaks at 2850–2950 cm^{-1} and 1050–1110 cm^{-1} derived from stretching vibration of —CH and asymmetric stretching vibration of Si—O—Si groups of the coupling agent appeared in GF-L- OH . Since the unreacted coupling agent was fully removed, the spectrum of GF-L- OH indicated that the coupling agent was successfully grafted onto the surface of GF *via* dehydration condensation. At the same time, the absorption peak at 867 cm^{-1} of GF-L- OH corresponding to the Si—OH was weaker than those of the other two types of glass fibers, indicating the lower —OH concentration on the surface of the fibers.

Figure 2(a) shows the XPS spectra of three types of GF. The major peak of all samples with the binding energy (BE) of 532.6 eV was assigned to O1s element signal while the minor

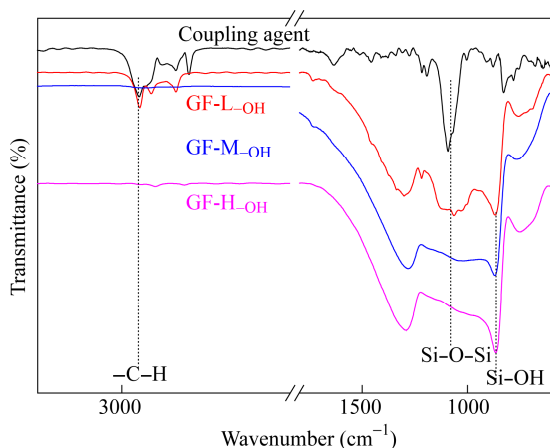


Fig. 1 FTIR spectra of the silane coupling agent, GF-L-OH, GF-M-OH and GF-H-OH

bands of 155.1 and 103.9 eV corresponded to Si2s and Si2p signals respectively, confirming the existence of Si—OH on the surface of glass fibers. Furthermore, the peak with the BE of 285.3 eV was assigned to C element signal, indicating the existence of Si—C on the surface of glass fiber. As demonstrated in Figs. 2(b) and 2(d), the C1s and Si2p peaks of GF-L-OH turned stronger than those of GF-M-OH and GF-H-OH, suggesting the surface of glass fiber was coated with a layer of coupling agent. In Fig. 2(c), obviously, the O1s signal of GF-H-OH had an increased BE, as compared with those of GF-M-OH and GF-L-OH, which indicated that there were more hydroxyl groups on the surface of GF-H-OH than GF-M-OH.

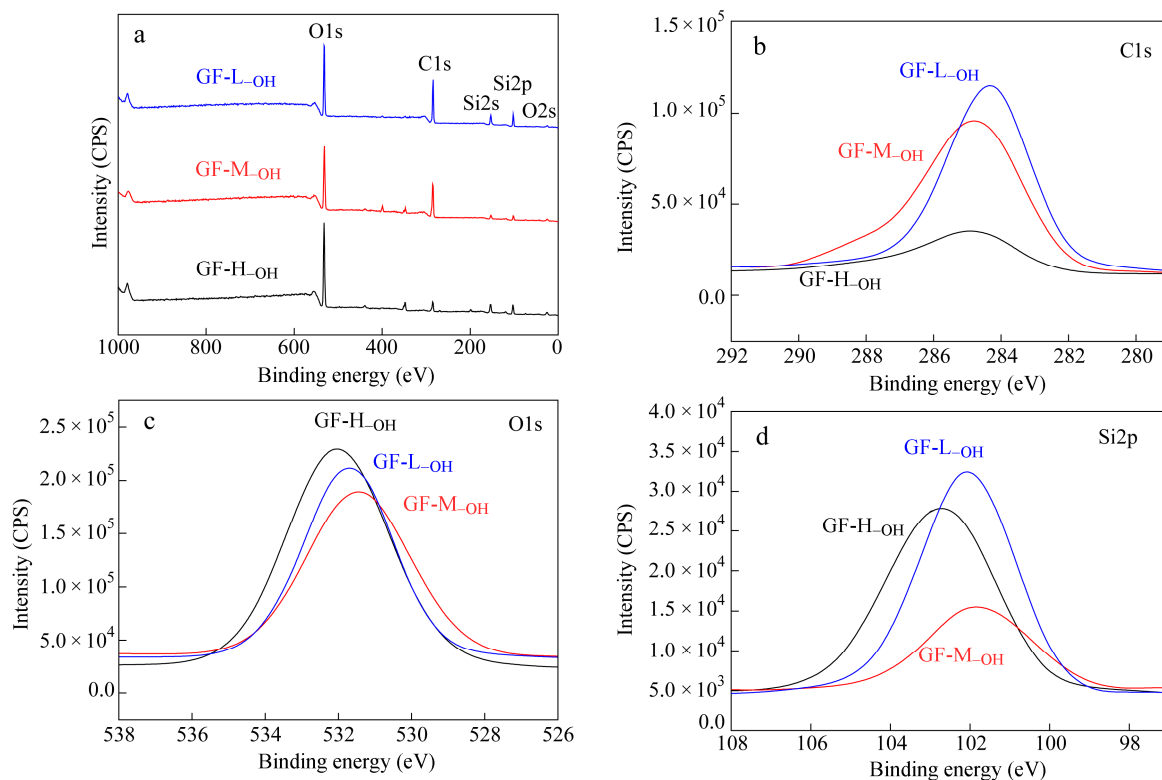


Fig. 2 XPS spectra of GF-L-OH, GF-M-OH, and GF-H-OH: (a) the survey scan spectra, (b) the C1s, (c) the O1s and (d) the Si2p core-level spectra

The various surface properties of three types of GF were also verified by TGA, as shown in Fig. 3. The fact that GF-L-OH shows the largest weight loss indicates that the silane coupling agent has reacted with—OH groups and the GF has the lowest OH concentrations on the surface of the GF.

Characterization of Interactions between Li-TFSI and Glass Fiber

Figure 4(a) shows the FTIR spectra of Li-TFSI and the three types of glass fibers with the surfaces coated by Li-TFSI. The intense absorption peaks at 1062, 1142, 1197 and 1331 cm^{-1} are assigned to the asymmetric stretching vibration of S—N—S, symmetric stretching vibration of O=S=O, symmetric bending vibration of CF_3 and asymmetric stretching vibration of O=S=O in TFSI[−], respectively^[24, 25]. GF-H-OH/Li-TFSI displays an absorption peak at 1204 cm^{-1} , attributed to symmetric bending vibration of CF_3 , which is higher than 1202 cm^{-1} for GF-M-OH/Li-TFSI and 1199 cm^{-1} for GF-L-OH/Li-TFSI. Such higher wavenumber shifting indicated that TFSI[−] has gradually increased interactions with Si—OH groups with increasing —OH concentrations on the surface of GF. Such interaction can be attributed to the hydrogen-bonding between the CF_3 and S=O groups of TFSI[−] with the Si—OH groups of GF. These results are consistent with results in FTIR, XPS and TGA. Moreover, it was also found that the absorption peak at 869 cm^{-1} assigned to Si—OH of the glass fiber shifted to lower wavenumber after surface coating with Li-TFSI (shown in Fig. 4b).

The different interactions between the Li-TFSI with the various types of GF can also be revealed by the SEM

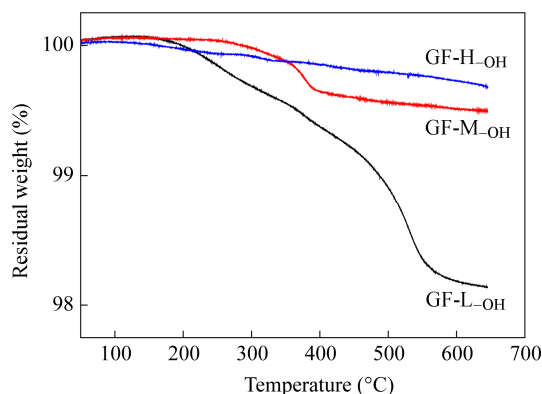


Fig. 3 TGA curves for GF-L-OH, GF-M-OH and GF-H-OH

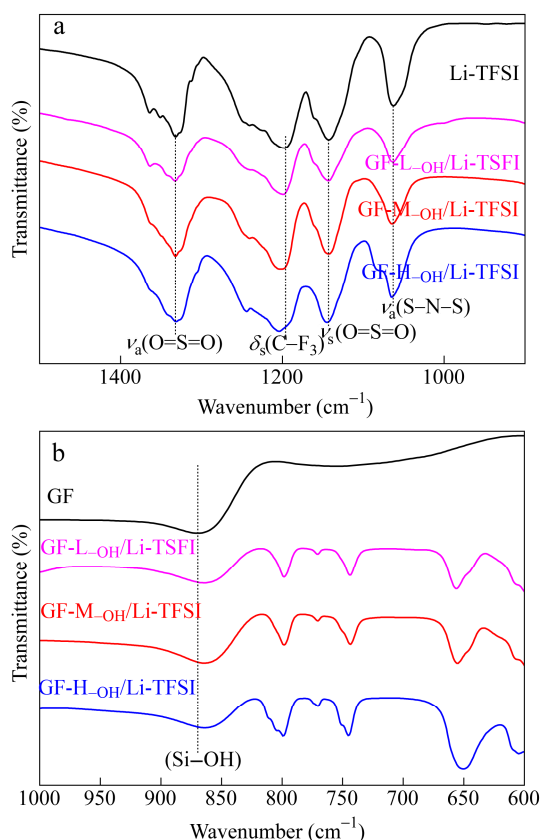


Fig. 4 FTIR spectra of (a) Li-TFSI, GF-L-OH/Li-TFSI, GF-M-OH/Li-TFSI and GF-H-OH/Li-TFSI and (b) GF, GF-L-OH/Li-TFSI, GF-M-OH/Li-TFSI and GF-H-OH/Li-TFSI

observation on the binary PP/Li-TFSI composites and the ternary PP/GF/Li-TFSI composites. As shown in Fig. 5(a), numerous Li-TFSI domains can be observed in the binary PP/Li-TFSI composites due to the immiscibility between PP and Li-TFSI. With incorporation of GF into the binary system, both the domain number and domain size of the ternary composites decreased obviously as compared with the binary PP/Li-TFSI blend. This is attributed to the interactions between GF and Li-TFSI. Parts of Li-TFSI were absorbed on the GF surface. Moreover, it was also found that few domains were observed for the PP/GF-H-OH/Li-TFSI (70/30/0.7) samples, indicating that the largest amount of Li-TFSI was absorbed on the surface of GF due to the highest

—OH concentrations on the GF surface.

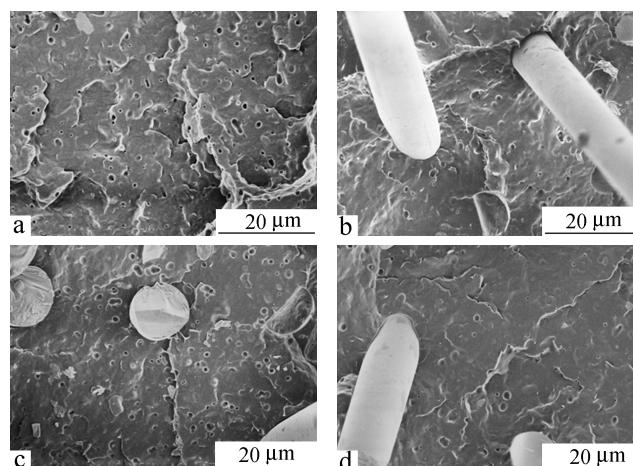


Fig. 5 SEM images of cross-fracture surface of (a) PP/ Li-TFSI (100/1), (b) PP/GF-L-OH/Li-TFSI (70/30/0.7), (c) PP/GF-M-OH/Li-TFSI (70/30/0.7), and (d) PP/GF-H-OH/Li-TFSI (70/30/0.7)

Effects of Different Interactions between GF and Li-TFSI on Antistatic and Mechanical Properties of PP/GF/Li-TFSI Composites

Figure 6 shows the electrical conductivity (black bars) and tensile strength (white bars) of PP/GF/Li-TFSI (70/30/0.7) composites as a function of various types of GF. PP/GF-M-OH/Li-TFSI composites have the highest electrical conductivity of $1.71 \times 10^{-10} \text{ S}\cdot\text{cm}^{-1}$. In contrast, the PP/GF-L-OH/Li-TFSI composite has an electrical conductivity of $2.71 \times 10^{-11} \text{ S}\cdot\text{cm}^{-1}$ and the electrical conductivity value is about $10^{-13} \text{ S}\cdot\text{cm}^{-1}$ for the PP/GF-H-OH/Li-TFSI composite. It is considered that the highest electrical conductivity can be achieved with the intermediate interactions between the Li-TFSI with GF. The weak interactions between GF-L-OH and Li-TFSI break the effective electrical conductive pathway formed by Li-TFSI on the surface of GF. On the other hand, the strong interaction would ‘fix’ the Li-TFSI on the surface of glass fiber, which leads to the great decrease in the electrical conductivity. Figure 6 also shows the tensile strength of the three composites. One can find that PP/GF-M-OH/Li-TFSI composite has the highest tensile strength of 39 MPa, while that of PP/GF-H-OH/Li-TFSI composite is only 29 MPa.

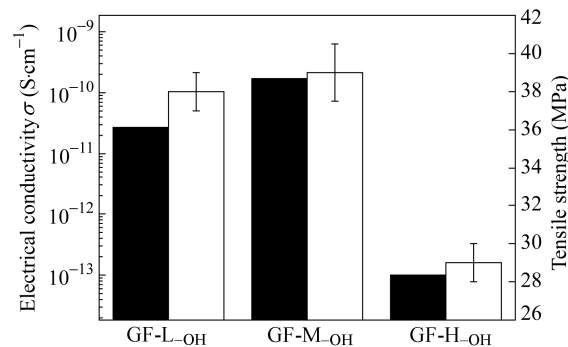


Fig. 6 Electrical conductivity (black bars) and tensile strength (white bars) of PP/GF/Li-TFSI (70/30/0.7) composites with indicated GFs

We have used OM measurement (Fig. 7) to elucidate the differences in the mechanical properties. Obviously, compared to the other two composites (Figs. 7a and 7b), the glass fibers show decreased length in Fig. 7(c). This might be originated from the treatment during the ignition in muffle furnace. Such condition breaks the integrity of GF network. Both factors lead to the rapid decrease of antistatic properties of PP/GF-H-OH/Li-TFSI composites. The tensile strength of PP/GF (70/30) composite is 31 MPa. A small amount of Li-TFSI incorporated into PP/GF-M-OH and PP/GF-L-OH composites results in increased tensile strength. This indicates the interfacial adhesion improvement of Li-TFSI. It is worth noting that the PP/GF-L-OH/Li-TFSI composite has almost similar tensile strength to that of PP/GF-M-OH/Li-TFSI composites. Further, 30% of GF is percolating in the PP matrix. Therefore, it is considered that the pathways for the ionic conduction are almost the same for the two types of GF with different lengths. The difference in the ionic conductivity is only originated from the interactions between the OH groups and ions. Besides, the mechanical properties should much depend on the length of the fibers. Therefore, we observed the low mechanical strength for the PP/GF-H-OH/Li-TFSI composite.

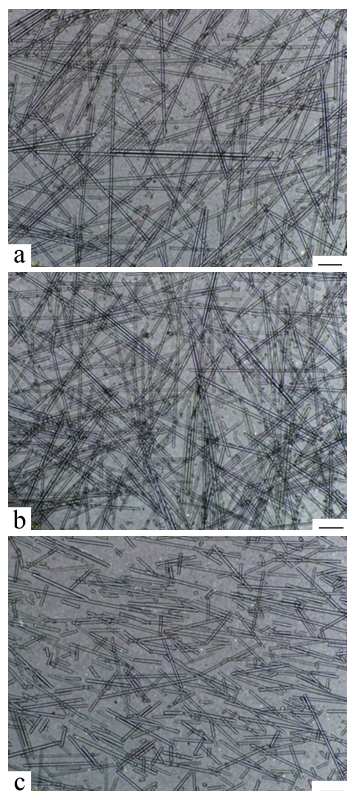


Fig. 7 OM images of (a) PP/GF-L-OH/Li-TFSI (70/30/0.7), (b) PP/GF-M-OH/Li-TFSI (70/30/0.7), (c) PP/GF-H-OH/Li-TFSI (70/30/0.7) (The scale bar is 100 μm .)

Discussion

It is very interesting to find that the surface properties of GF affect the antistatic properties of PP/GF/Li-TFSI composites. The —OH group concentration of GF changes the specific interactions between GF and Li-TFSI. As the antistatic performance of PP/GF/Li-TFSI composites is attributed to

the ion movements through the pathways provided by GF network, which has been demonstrated in the previous work, it is therefore important to elucidate the mechanism of ion transport in PP/GF/Li-TFSI composites. Li^+ cations have been reported to take the functions for charge transport in many ionic liquid-based gel polymer electrolyte investigations^[26, 27]. Borodin *et al.*^[27] reported the mechanism of ion transport in amorphous poly(ethylene oxide)/Li-TFSI and investigations on the ion transport mechanism by molecular dynamic simulations. Both Li^+ and TFSI^- have been confirmed to be coupled to the polymer segmental relaxation. In their simulations, they observed various changes in Li^+ coordination with time that potentially could contribute to Li^+ transport. One of the events was Li^+ decomplexing from PEO segments and complexing by TFSI^- anions (without PEO) with a subsequent jump to another PEO segment within a few nanoseconds under external field. Moreover, they also found a TFSI^- anion approaching a Li^+ cation at the moment when the cation undergoes intersegmental jump, indicating that PEO segments and TFSI^- both play important roles in charge transport of Li^+ . In the present work, we considered that the strong Lewis acid-base interaction between cation and electron-donating oxygen atoms of —OH in GF would confine the movement of Li^+ . So the single effects of GF would not contribute to the charge transport. The only way for Li^+ to act as a charge carrier is that Li^+ hops from one O^- to another bridged by TFSI^- . Li^+ ions certainly have electrostatic interaction with TFSI^- . So it is rational that TFSI^- has important effects on the charge transport of Li^+ . For glass fiber, the polymer segmental relaxation of PEO could be replaced by swinging of the hydroxyl groups on the surface of GF. Such sequential “complexing and decomplexing” effects between O^- and TFSI^- contributed to the Li^+ transport. Figure 8 shows the schematic diagram of Li-TFSI transfer mode on glass fiber network. GF forms nice network as the pathways for the movement of ions. The final conductivity is then dependent on the interactions between the ions with —OH groups of GF. The interactions between TFSI^- /GF-H-OH are very strong due to the high concentration of —OH groups on the GF surface; the movement of TFSI^- may be confined by the interactions, so the TFSI^- would not approach Li^+ at the moment that Li^+ decomplexing from O^- . Therefore, the poor antistatic properties have been observed for PP/GF-H-OH/Li-TFSI composite. On the other hand, the weak interactions between GF-L-OH and Li-TFSI lead to the free Li-TFSI in the PP matrix, so the ion concentration on the GF surface is low. Therefore, the conductivity for the PP/GH-L-OH /Li-TFSI is also low.

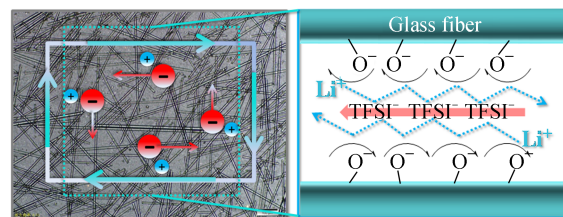


Fig. 8 Schematic diagram of Li-TFSI transfer mode on glass fiber network

CONCLUSIONS

In this work, three types of glass fibers with different —OH group concentrations have been prepared. It was found that the —OH group concentrations on the surface of GF had significant effects on the interaction between Li-TFSI and glass fiber, which consequently influenced the antistatic and mechanical properties of PP/GF/Li-TFSI composites. The glass fiber with high —OH concentrations could confine the movement of ions, which decreased the antistatic properties of PP/GF/Li-TFSI composites.

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