

## Conductive Performances of Elastomeric Electrolytes Based on Maleic Anhydride-*g*-poly(1, 2-butadiene) Grafting Polyethylene Glycol 400 and LiClO<sub>4</sub>

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**ABSTRACT:** Elastomeric electrolyte films consisting of maleic anhydride-*g*-poly (1, 2-butadiene) (MAPB) grafting polyethylene glycol 400 (PEG<sub>400</sub>) as matrix and LiClO<sub>4</sub> as alkali salt at the mole ratio of [O]: [Li<sup>+</sup>] at 6, 8, and 10, respectively are prepared. An equivalent circuit is proposed based on the electronic impedance spectroscopy plots by using the metal/electrolyte film/metal model, the simulant plots are found matched well with the tested plots, and the conductivity variations with salt concentrations and temperatures are calculated accordingly. The film with the mole ratio of [O]: [Li<sup>+</sup>] at 8:1 exhibits higher conductivity at room temperature, due to its relatively stronger dissociation of oxygen atom to LiClO<sub>4</sub> as supported by the FTIR spectroscopy. Their conductivity dependent on temperatures obeys the Arrhenius equation from 303 to 363 K, while it keeps almost unchanged at higher temperatures; these changes are found associate with the strong complexation of oxygen atom with Li<sup>+</sup> and the state transference of MAPB elastomer as observed by the DSC analysis. Besides, these films are still thermally stable at around 200°C as observed by the TGA analysis and may be promising solid polymer electrolytes with high stability. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

**KEYWORDS:** electrochemistry; elastomers; thermal properties

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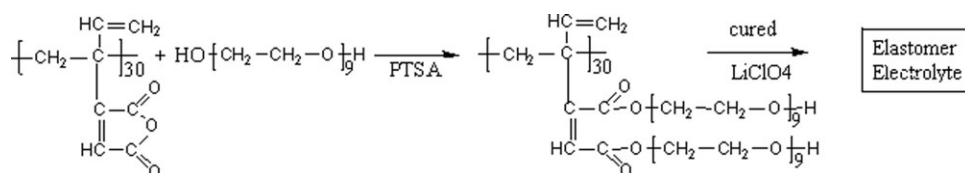
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### INTRODUCTION

Solid polymer electrolytes (SPEs) consisting of polymer and Li salt also called ion-conductive polymer has become a promising material in the area of lithium polymer batteries as it not only has the flexibility, favorable film, stability, light weight, low cost but also high mechanical strength, no leakage of electrolyte and improved safety.<sup>1,2</sup> Since Wright et al.<sup>3</sup> found that poly (ethylene oxide) (PEO) dissolved salts had an ionic conductivity of  $10^{-8} - 10^{-7} \text{ S cm}^{-1}$  at ambient temperature, the high molecular weight poly (ethylene oxide) (PEO) is still one of the most frequently used polymer matrix because of its solvation effect, complexation ability and ion transport mechanism directly connected with the alkaline salt (Li<sup>+</sup>).<sup>4–6</sup> Besides, consistent research efforts have been devoted to preparing SPEs by using other polymers.<sup>7–9</sup> However, almost all of these SPEs showed low ion conductivity and high interfacial resistance at ambient temperature due to their chemical or physical characteristics which restricted the mobility of ions.

To improve the mobility of polymer chains, organic plasticizers have been by far the most frequently incorporating agents used

in ionic polymer electrolytes. Various organic compounds or low molecular weight polymers such as propylene carbonate (PC), ethylene carbonate (EC), polyethylene glycol (PEG), etc., have been reported having effective plasticizing functions.<sup>10,11</sup> Nano-fillers such as nano-Al<sub>2</sub>O<sub>3</sub>, nano-SiO<sub>2</sub>, and so on have also been found effective in improving the conductivity of SPEs.<sup>12</sup> However, the improvement in conductivity is rather limited by the addition of either plasticizers or nano-fillers. As such, some researchers tried to add plasticizer together with nano-filler to improve the conductivity of SPEs. For example, Pitawala et al.<sup>13</sup> demonstrated that the incorporation of 50 wt % ethylene carbonate (EC) and 15 wt % nano-Al<sub>2</sub>O<sub>3</sub> to the (PEO)<sub>9</sub>-LiTf electrolytes showed a significant enhancement in conductivity while retaining the mechanical strength of electrolyte films. The conductivity of SPEs is also found greatly affected by the type and concentration of ionic salts used, and often fluctuates with temperatures, while the conclusive mechanisms are still unclear.<sup>14–16</sup> Besides, the essential path is still developing novel polymers in terms of the structure design. For example, by copolymerization of acrylonitrile and poly (ethylene glycol-methyl methacrylate) (PEGMEM), a solid electrolyte



**Scheme 1.** The schematic illustration of MAPB-PEG<sub>400</sub> formation.

based on comb-like copolymer (AN-*co*-PEGMEM) could be prepared with higher conductivity than that of PAN base system.<sup>17</sup> By photo-induced polymerization, poly(ethylene glycol) (PEG) methacrylate macromonomers was grafted onto poly(methyl methacrylate) (PMMA), and the conductivity of these solid electrolytes based on PEG-grafted PMMA was found dependent on the PEG contents.<sup>18</sup>

As a modified elastomeric material, maleic anhydride-*g*-poly (1, 2-butadiene) (MAPB) was used to prepared high performance coating with excellent tenacity and anticorrosive ability in one of our previous studies.<sup>19</sup> Its flexibility, relaxation, and mechanical strength make it a suitable candidate for solid polymer electrolyte in terms of polymer structure design, in case its structure combines the elastomeric relaxation and the polar complex together, which may bring some interesting results in this field. As such, in this study, a novel elastomeric electrolyte based on maleic anhydride-*g*-poly (1, 2-butadiene) (MAPB) grafting long branched polyethylene glycol 400 (PEG<sub>400</sub>) chains as matrix and LiClO<sub>4</sub> as alkali salt was prepared. Their conductivity changes with the concentrations of Li salt and temperatures were investigated by electronic impedance measurement, and these changes related to the structures of MAPB-PEG<sub>400</sub>, the interaction of the components and the stability of SPEs were investigated by using Fourier transform infrared (FTIR) spectra, differential scanning calorimetry (DSC), and thermo-gravimetric analysis (TGA) measurements.

## EXPERIMENTAL

### Reagents

Maleic anhydride-*g*-poly (1, 2-butadiene) (MAPB) (maleic anhydride 15 wt % of MAPB, LPB terminated by xylene,  $M_n = 1400$ , number of structure poly-butadiene units  $n = 30$ , CR), provided by Yan-shan Petrochemical Corp., Sinopec, China. Lithium perchlorate trihydrate (LiClO<sub>4</sub>·3H<sub>2</sub>O, AR), dried at 120°C under vacuum for 2 h before use; *p*-toluenesulfonic acid (PTSA, AR), used as received; dicumyl peroxide (DCP, CR), used as received, these chemicals were purchased from Sino-pharm Chemical Reagent, China. Polyethylene glycol (PEG400, AR), used as received; Chloroform (CHCl<sub>3</sub>, AR), used as received; Acetone (C<sub>3</sub>H<sub>6</sub>O, AR), used as received; Benzoyl peroxide (BPO, CR), used as received; these chemicals were purchased from Beijing Chemical Reagent Company, China.

### Preparation of Polymer Electrolytes Films

**The Graft MAPB with PEG<sub>400</sub>.** MAPB was mixed with PEG<sub>400</sub> (the mole ratio of maleic anhydride and polyethylene glycol is 1:1) in a 250-mL flask fitted with a reflux condenser and a water segregator under magnetic force stirring. PTSA (3% of the mass of maleic anhydride) as a catalyst was dissolved in 110

mL CHCl<sub>3</sub>,<sup>20</sup> and then added into the above flask. By purging nitrogen as an inert atmosphere, the mixture was heated to a constant temperature (62°C) in a water bath with continuous stirring. After it was refluxed for 9 h, a light brown viscous solution was obtained. The formation of MAPB-PEG<sub>400</sub> is plotted as shown in Scheme 1.

**Preparation of Polymer Electrolytes Film.** LiClO<sub>4</sub>, DCP and BPO were dissolved in acetone under manual stirring to obtain a clear solution. MAPB-PEG<sub>400</sub> was added under continuous stirring, then the mixture was put in the ultrasonic cleaners for about 30 min to get a transparent solution (the masses of MAPB-PEG<sub>400</sub> with polyethylene glycol was calculated based on mole ratio of [O] and [Li<sup>+</sup>], where [O] represents the mole number of oxygen atoms from carbonyl and ether groups after maleic anhydride reacting with ethylene glycol as shown in Scheme 1, [Li<sup>+</sup>] represents the mole number of Li<sup>+</sup> in LiClO<sub>4</sub> salt). Three samples with [O]: [Li<sup>+</sup>] at 6:1, 8:1 and 10:1 were prepared, respectively. Afterwards, these mixtures were heated to 70°C and maintained for about 1 h to evaporate the acetone and chloroform. The above slurry polymer electrolytes were spread into a rectangular aluminum mould, and then dried in a vacuum oven at 80°C for 24 h to remove the residual solvent and achieve pre-curing. Finally, the samples were heated to 180°C for 30 min for post-curing,<sup>19</sup> to obtain elastic polymer electrolytes films with thicknesses about 0.7–0.9 mm.

### Characterization

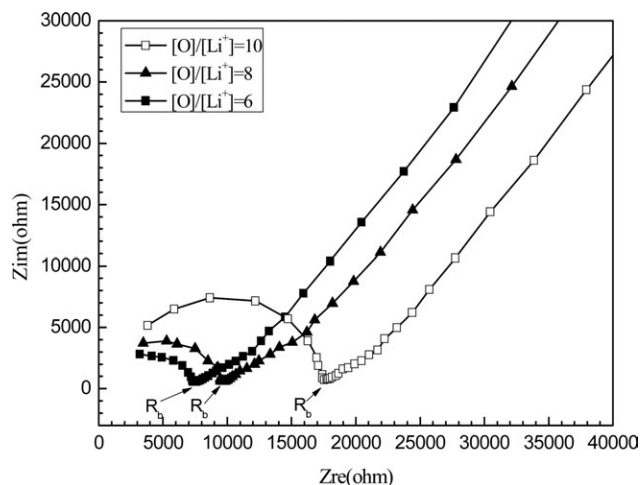
Fourier transform infrared spectroscopy (FTIR): FTIR spectra were performed by using FTs-3000 (DIGILAB, America) with a resolution of 0.1 cm<sup>-1</sup> in the range of 400–4000 cm<sup>-1</sup>.

**Differential Scanning Calorimetry.** DSC curves were recorded by using Diamond DSC (PerkinElmer, America), at a heating rate of 10°C min<sup>-1</sup> from -50°C to 150°C under nitrogen.

**Thermo-Gravimetric Analysis.** Thermo-gravimetric analysis (TGA) curves were measured by using STA449F3 (NETZSCH, German) from 50 to 480°C at a heating rate of 10°C min<sup>-1</sup> under nitrogen.

### Electronic Impedance Spectroscopy

Electronic impedance spectroscopy (EIS) plots were measured by using the Versa STAT MC electrochemical station (EG & G, Inc., USA) in an electrochemical cell consisting of the electrolyte film sandwiched between two stainless steel electrodes. The measurements were carried out over a frequency range of 0.1–10<sup>6</sup> Hz. The sample for the ionic conductivity measurement had a square shape with 1 cm in length of side. The electronic impedance plots were matched with ZSimpWin software under a fitted equivalent circuit. The ionic conductivity ( $\sigma$ ) of films was calculated by the equation:  $\sigma = d/(R_f \cdot S)$ ,<sup>10</sup>



**Figure 1.** EIS plots of MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films ([O]:[Li<sup>+</sup>] = 6:1, 8:1, 10:1) under room temperature.

where  $\sigma$  is the conductivity,  $R_b$  is the bulk resistance from EIS plots,  $d$  is the thickness of films, and  $S$  is the contact area ( $S = 1 \text{ cm}^2$ ) between the stainless electrodes and solid MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> electrolytes film. When the conductivity was measured at certain higher temperature, the above cell had to be put in a cabin which was immersed in a circulating oil bath to maintain that temperature for 10 min, and then performed the EIS measurement.

## RESULTS AND DISCUSSION

### EIS Plots of the Solid Polymer Electrolytes Films

Figure 1 shows the EIS plots of the solid polymer electrolyte films with the mole ratio of [O]: [Li<sup>+</sup>] at 6:1, 8:1, 10:1 under room temperature, respectively. The diagrams are composed of compressive semicircle and a straight sloping line at low-frequency end. The sample bulk resistance ( $R_b$ ) was obtained from the intersection point of the semicircle with that of the spike.<sup>21</sup> In general, the size of the semicircle represents the resistance of the SPEs film; a bigger semicircle implies a larger resistance. The resistance of SPE film with mole ratio of [O]: [Li<sup>+</sup>] at 6:1 does not exhibit much difference from that of the film with mole ratio of [O]: [Li<sup>+</sup>] at 8:1. However, the resistance of SPE film with mole ratio of [O]: [Li<sup>+</sup>] at 10:1 is about two times bigger than that of the film with mole ratio of [O]: [Li<sup>+</sup>] at 8:1. This suggests that less amount of lithium salt results in the lack of sufficient conduction carrier,<sup>14</sup> while excessive amount of lithium salt decreases the mobility of polymer chain by the pseudo-cross-linking between Li<sup>+</sup> and oxygen atom in MAPB-PEG<sub>400</sub> chains.<sup>22</sup>

### EQUIVALENT CIRCUIT BASED ON METAL/SPEs/METAL CELL MODEL

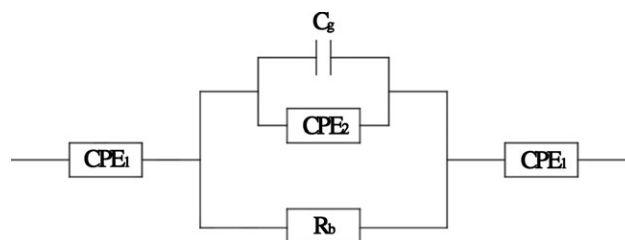
The EIS plots are important methods to study the dynamic behavior of electrode reaction and electrode interfacial phenomena. Usually, the SPEs film can be sandwiched between two metal electrodes to make metal/SPEs/metal cell model so that the equivalent circuit can be set up and the ion conductivity can be calculated.<sup>10</sup> Figure 2 shows the equivalent circuit based

on the SS/SPEs/SS blocking electrodes. In Figure 3,  $R_b$  is the bulk resistance of the cell, reflecting a combined resistance of the electrolyte, separator and electrodes;  $C_g$  represents the geometrical capacitance; CPE is the constant phase element<sup>23</sup> (the resistance of CPE component is showed as follows:  $Z_{CPE} = A(j\omega)^{-n}$  and  $\alpha = (1-n)\pi/2$ , where  $Z$  is the resistance of the capacitance;  $A$  is a constant about the frequency;  $\alpha$  is the angle between the straight sloping line at low-frequency and the vertical direction. The straight sloping line at low-frequency is perpendicular to the horizontal axis as  $n = 1$ , and CPE is an ideal capacitance; CPE is a resistance as  $n = 0$  and it is an inductor as  $n = -1$ . However,  $n$  is about 0-1 in our data fitting which implies CPE is made up of two components, resistance and capacitance in this study, so CPE<sub>1</sub> and CPE<sub>2</sub> correspond to the contact resistance and capacitance of unevenly contact interface between stainless electrodes and SPEs films<sup>24</sup>

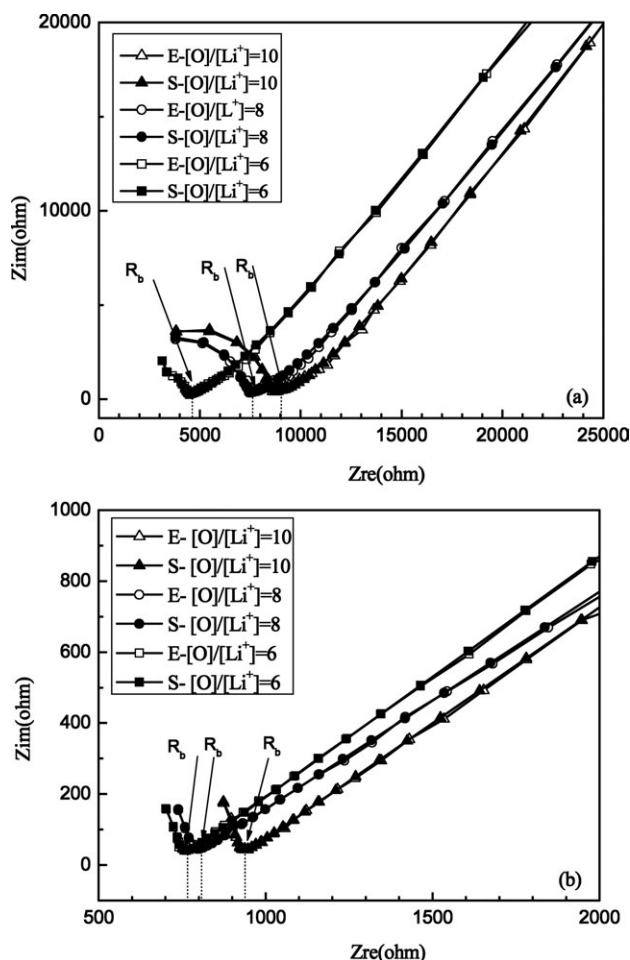
Figure 3 shows the EIS results of these MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films with the mole ratio of [O]: [Li<sup>+</sup>] at 6:1, 8:1 and 10:1 tested at 40 and 120°C, respectively. All the EIS plots of SPEs films with different mole ratios of [O]: [Li<sup>+</sup>] at different temperatures are composed of compressive semicircle at high frequency and a straight sloping line at low frequency end; at the same time, all the semicircle arcs gradually reduce as the temperature increases. The curves of simulant lines by ZSimpWin software with the equivalent circuit (as shown in Figure 2) and the experimental curves match well in the testing range regardless of temperatures, indicating the rationality of the equivalent circuit. On the basis of the stimulant EIS plots, the conductivity could be calculated with the equation of  $\sigma = d/(R_b \cdot S)$ .<sup>10</sup>

### Temperature Dependence of Conductivity

Figure 4 shows the conductivity dependence on temperatures for MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films with mole ratio of [O]: [Li<sup>+</sup>] at 6:1, 8:1, and 10:1, respectively in the temperature range from 303 to 393 K. The conductivity of all the samples increases almost linearly with temperatures at the lower temperature range from 303 to 363 K which can be described by Arrhenius relationships as mostly reported in the literatures.<sup>24-26</sup> This conductivity growth with the increase in temperatures from 303 to 363 K is due to the improved mobility of MAPB-PEG<sub>400</sub> chains and the enhanced interaction between oxygen atom and Li<sup>+</sup>, resulting in the much more rapid translocation of Li<sup>+</sup> at elevating temperatures. However, when the temperature is above 363 till 393 K in our testing ranges, their conductivity keeps almost unchanged, and their relationships no longer follow Arrhenius equation, the reason for this will be discussed in the followings.



**Figure 2.** Equivalent circuit based on SS/MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> film/SS blocking electrodes model.



**Figure 3.** Experimental (*E*) and stimulant (*S*) EIS plots of the MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films ([O]: [Li<sup>+</sup>] = 6:1, 8:1, 10:1) at differential temperatures [(a) 40°C (b) 120°C].

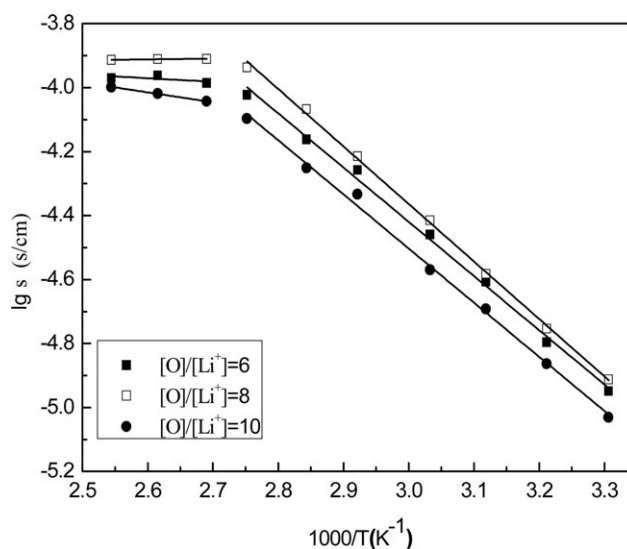
### Motivation of Lithium Ion Movement

Dissociation of the lithium salt is an important issue that determines the ionic conductivity of the SPEs film, and the degree of dissociation by polymer segments can be evaluated by FTIR. Figure 6 shows the FTIR spectra of MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films with mole ratio of [O]: [Li<sup>+</sup>] at 6:1, 8:1, and 10:1, respectively in the range of 580–660 cm<sup>-1</sup>. The peak at 624 cm<sup>-1</sup> can be ascribed to free ClO<sub>4</sub><sup>-</sup> and the peak at 637 cm<sup>-1</sup> can be ascribed to ion-pair formation or contact of ClO<sub>4</sub><sup>-</sup> with Li<sup>+</sup>, similar to those LiClO<sub>4</sub> in poly (AN-co-PEG-MEM)/PC as reported by Liang et al.<sup>17</sup> and LiClO<sub>4</sub> in SN: PAN composite electrolyte film as reported by Patel et al.<sup>27</sup> The corresponding intensity ratio of free ClO<sub>4</sub><sup>-</sup> to ion-pair for SPE with different ratio of [O]: [Li<sup>+</sup>] is in the order of 8:1 > 6:1 > 10:1, suggesting that more free Li<sup>+</sup> ions exist in sample with [O]: [Li<sup>+</sup>] at 8:1, and hence higher conductivity at room temperature is observed from Figures 2 and 4. It is interesting that lower salt concentration does not imply higher amount of free ions, the special structure of MAPB-PEG<sub>400</sub> may account for this; It may be inferred that when MAPB was cured, some neutral salts were isolated by the cross-linked nonpolar butadiene chains and the possibility of their interaction with oxygen

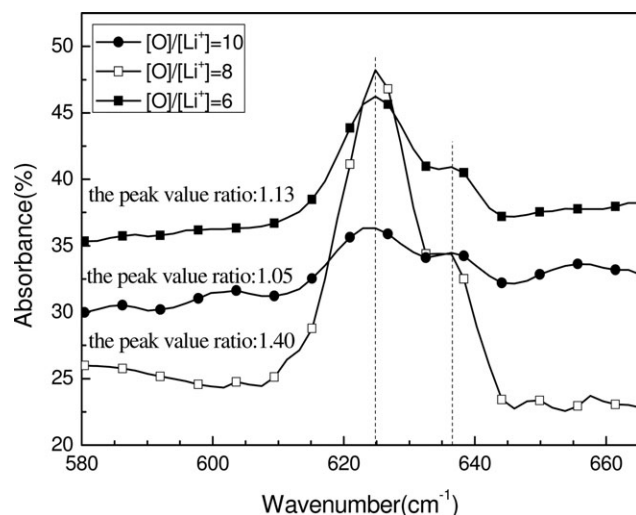
was reduced in case of low salt amount, as such the amount of free ions decreased.

The DSC curves of MAPB-PEG<sub>400</sub> matrix and MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films are shown in Figure 6. The endothermic behavior of the MAPB-PEG<sub>400</sub> matrix is observed from -40 to -25°C corresponding to the range of glass transition temperatures (*T<sub>g</sub>*) which are pushed to a higher range from -30 to 0°C with the addition of LiClO<sub>4</sub>, indicating that the flexibility of the polymer chains decreases with the addition of LiClO<sub>4</sub> in the MAPB-PEG<sub>400</sub> matrix. This reduction in flexibility of polymer chains in SPEs is usually attributed to the coordinating sites of [O]: [Li<sup>+</sup>] and the ion-pairs restricting the mobility of the side chain segments.<sup>28,29</sup> Above these temperatures, the state of MAPB-PEG<sub>400</sub> changes to viscoelastic state in which the mobility of its segments increases with the increase in temperatures.<sup>30</sup> Under this circumstance, the faster movement of the polar segments promotes the dissociation of ion-pairs and the faster transportation of free ions, resulting in the increase in conductivity with temperature as shown in Figure 4.

In general, the state transition from viscoelastic to highly deformed elastic is an endothermic transversion with slow energy consumption for almost all the polymers,<sup>32</sup> and the broad hump for MAPB-PEG<sub>400</sub> implies its conformity with this principle. However, an obvious exothermic peak is found in the temperature range of 80–110°C centered at around 90°C for these SPEs films in Figure 6, suggesting that the addition of lithium salt is responsible for this. The conductive mechanism of the solid polymer electrolytes has been usually explained in terms of the dissociation of ion-pairs by polymer chains, the complexation between the polar atom of polymer chain with ions, and the transportation of ions.<sup>33</sup> During the state transition upon heating, the more and more aggressive movement of polymer chains and ions results in the higher and higher strong interaction between ions and the oxygen atoms, also the much stronger friction among polymer segments with ions, and both



**Figure 4.** Temperature dependence of the conductivity for MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films ([O]:[Li<sup>+</sup>] = 6:1,8:1,10:1).

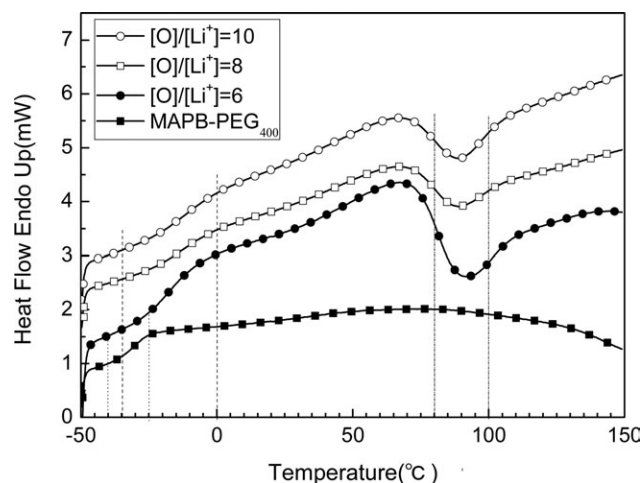


**Figure 5.** FTIR spectra of MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films ([O]: [Li<sup>+</sup>] = 6:1, 8:1, 10:1).<sup>17,27</sup>

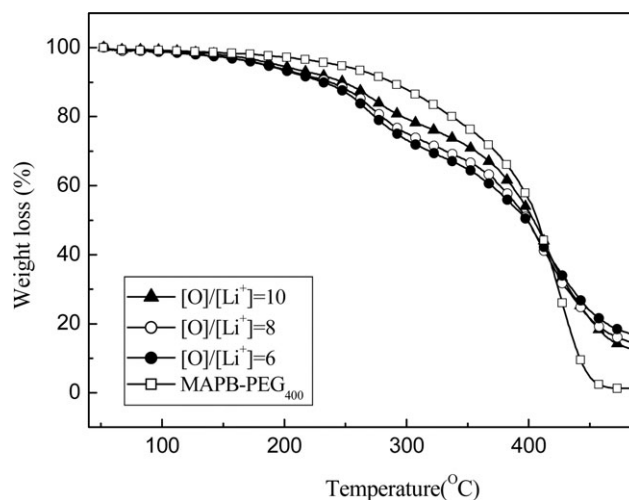
of these two functions are considered as exothermal effects.<sup>34</sup> Upon heating to much higher temperatures, the state of MAPB-PEG<sub>400</sub> changes to highly deformed elastic state in which all the polymer chains are in the most extended state and their mobility does not increase much with the increase in temperatures,<sup>31</sup> hence, the ion mobility promoted by side chains remains almost unchanged, resulting in their conductivity fluctuation in a very small range as shown in Figure 4.

### Thermal Gravimetric Analysis

The thermal stabilities of MAPB-PEG<sub>400</sub> matrix and MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> with mole ratio of [O]: [Li<sup>+</sup>] at 6:1, 8:1, and 10:1, respectively were measured by TGA as shown in Figure 7. MAPB-PEG<sub>400</sub> matrix exhibits a gradual degradation from 250 to 350°C, and then a rapid degradation till it is almost completely decomposed at about 480°C. However, the samples of MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> exhibit a trend of weight loss starting at about



**Figure 6.** Differential scanning calorimetry (DSC) curves of MAPB-PEG<sub>400</sub> matrix and MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films ([O]: [Li<sup>+</sup>] = 6:1, 8:1, 10:1).



**Figure 7.** TGA curves of MAPB-PEG<sub>400</sub> and MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> films ([O]: [Li<sup>+</sup>] = 6:1, 8:1, 10:1).

200°C, then a more gently gradual weight loss till 350°C, and finally a drastic weight loss till 480°C with residues around 15 wt %. The earlier decomposing temperature of MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> in comparison with MAPB-PEG<sub>400</sub> matrix films reveals that the addition of LiClO<sub>4</sub> decreases the thermal stability of the SPEs film. This may be attributed to the weakening of the ether (C—O) bond caused by the strong coordination between Li<sup>+</sup> ion and oxygen atom.<sup>2</sup> These results together with the conductivity and DSC results indicate that these MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> electrolytes still possess good thermal stability up to 200°C.

### CONCLUSIONS

MAPB grafting PEG<sub>400</sub> was used to prepare MAPB-PEG<sub>400</sub>/LiClO<sub>4</sub> elastomeric electrolytes films with the mole ratio of [O]: [Li<sup>+</sup>] at 6:1, 8:1 and 10:1 by solution-casting method. Their conductive performances associate with salt concentration and temperatures were found strongly dependent on the complexation between Li<sup>+</sup>, oxygen, and polymer chain movements. The bulk conductivity of the electrolytes films could reach 10<sup>-4</sup> S cm<sup>-1</sup> at room temperature and exhibited linear increase with the increase in temperature from 303 to 363 K following Arrhenius equations, resulted from the enhancement in the dissociation of neutral salt by the polar side chains, the interaction between Li<sup>+</sup> and oxygen atoms and the mobility of the side chains upon heating. While their conductivity kept almost unchanged upon continuously heating above 363 K, resulted from the utmost moving ability of the modified elastomer after its state transversion and the their interactions with ions in a maximum level. Their decomposition temperature at above 200°C suggests that they may be good choices to enhance the practical performance as a solid polymer electrolyte.

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