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# Polyethylene-block-Polyacrylate Block Copolymer Electrolytes with High Room-Temperature Ionic Conductivity

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## Abstract

Conventional block copolymer (BCP) based polymer electrolytes take advantage of the mechanical strength provided by a high glass transition temperature (T<sub>g</sub>) block, and the ion-conducting properties of a low T<sub>g</sub> block. However, the presence of a glassy phase often limits conductivity by suppressing segmental mobility. In this work, we develop polyethylene-block-poly(meth)acrylate block copolymers doped with ionic liquid 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (EMIM-TFSI) and achieve a significantly high ionic conductivity of ~1 mS/cm at room temperature. The high conductivity is attributed to the low T<sub>g</sub> of both blocks which leads to enhanced chain dynamics and efficient ion transport, coupled with a sharp interface between PE and PMMA blocks and crystallinity of the PE block. At the same time, our mechanical measurements show that these materials retain elastic solid-like behavior, providing the mechanical stability required for solid electrolyte applications. We also studied the effects of nanoscale morphology on ionic transport for a better understanding of the high ionic conductivity of these systems. Our study indicates that nanoscale interfacial structure and segmental chain motion jointly contribute to the elevated ionic conductivity of these materials. Overall, these polyethylene-block-poly(meth)acrylate electrolytes represent a promising route toward mechanically robust, thermally stable, and highly conductive solid-state electrolytes.

## Keywords

Polymer Electrolytes, Ionic Conductivity, Lithium Batteries, ionic liquid

# Polyethylene-*block*-Polyacrylate Block Copolymer Electrolytes with High Room-Temperature Ionic Conductivity

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**KEYWORDS:** *Polymer Electrolytes, Ionic Conductivity, Lithium Batteries, ionic liquid*

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**ABSTRACT:** Conventional block copolymer (BCP) based polymer electrolytes take advantage of the mechanical strength provided by a high glass transition temperature ( $T_g$ ) block, and the ion-conducting properties of a low  $T_g$  block. However, the presence of a glassy phase often limits conductivity by suppressing segmental mobility. In this work, we develop polyethylene-*block*-poly(meth)acrylate block copolymers doped with ionic liquid 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (EMIM-TFSI) and achieve a significantly high ionic conductivity of  $\sim 1$  mS/cm at room temperature. The high conductivity is attributed to the low  $T_g$  of both blocks which leads to enhanced chain dynamics and efficient ion transport, coupled with a sharp interface between PE and PMMA blocks and crystallinity of the PE block. At the same time, our mechanical measurements show that these materials retain elastic solid-like behavior, providing the mechanical stability required for solid electrolyte applications. We also studied the effects of nanoscale morphology on ionic transport for a better understanding of the high ionic conductivity of these systems. Our study indicates that nanoscale interfacial structure and segmental chain motion jointly contribute to the elevated ionic conductivity of these materials. Overall, these polyethylene-*block*-poly(meth)acrylate electrolytes represent a promising route toward mechanically robust, thermally stable, and highly conductive solid-state electrolytes.

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## INTRODUCTION

Solid-state polymer electrolytes (SPE) offer advantages over traditional organic liquid electrolytes in lithium batteries due to their low flammability, flexibility, and mechanical robustness.<sup>1, 2</sup> Specifically, SPEs such as poly(ethylene oxide) (PEO) have been investigated as materials to replace conventional liquid electrolytes since their oxygen atoms can efficiently promote ion conduction. However, their crystallinity at room temperature leads to poor ionic conductivity and cycling stability.<sup>3, 4</sup> To modulate the ionic conductivity, the addition of polymers to create nanostructured conductive polymer blends has been investigated, with the goal that structural features fine-tune the morphology and architecture of the polymers and blends to enhance their mechanical strength and ionic conductivity.<sup>5-7</sup> Nanostructured SPEs in the form of block co-polymers (BCPs) can self-assemble into ordered structures by block incompatibility, and they also can help to decouple mechanical and electrochemical properties. These “nanostructured electrolytes”, which contain segments of PEO, can confine ion transport and metal growth within nanoscale domains, thereby

reducing the likelihood of dendritic growth via uniform Li deposition.<sup>8,9</sup> For example, polystyrene-*block*-poly(ethylene oxide) copolymers, PS-*b*-PEO, are one of the most investigated block copolymers, in which the PEO is the conducting component, and the PS is the glassy component to give mechanical strength. Although the ionic conductivity can be enhanced by adding different concentrations of lithium bis(trifluoromethanesulfonyl)imide (LiTFSI) salt to the polymer matrix and decreasing grain growth, the ionic conductivity is only high at elevated temperatures (80-100 °C) due to the high melting transition temperature ( $T_m$ ) of the conductive phase<sup>10</sup>, and a low (ambient) temperature ionic conductivity remains an obstacle. Alternative BCPs such as polystyrene-*block*-poly(methyl methacrylate) (PS-*b*-PMMA) copolymers have been tested as well, but their ionic conductivity at room temperature is not significantly higher, with the highest conductivity ( $2 \times 10^{-5}$  S/cm) achieved for a PS-*b*-PMMA BCP with a cylindrical phase, with an IL to MMA mole ratio of  $r=0.45$ .<sup>11</sup>

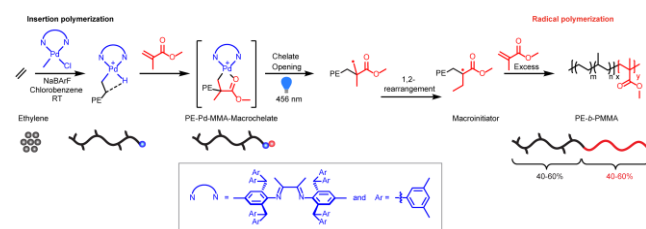
SPEs are still a promising class of electrolyte materials; however, these data indicate that their broader application

has so far been limited by insufficient room-temperature ionic conductivity and an incomplete understanding of the structural and dynamical factors that govern ion transport. It is now better understood that improving room-temperature ionic conductivity is the matter of control over polymer chemistry, monomer composition, and block-copolymer architecture, since these parameters strongly influence segmental mobility, crystallinity, and nanoscale ion-transport pathways.<sup>12,13</sup>

In this work, we report a new class of block copolymer electrolytes based on polyethylene-block-poly(methyl methacrylate), PE-*b*-PMMA, in which mechanical support is provided by the semicrystalline PE in comparison to the conventional glassy structural block. The ionic conductivity was investigated as a function of ionic-liquid content, and the PE-*b*-PMMA/EMIM-TFSI electrolytes reached  $\sim 1$  mS/cm at room temperature, which is more than an order of magnitude higher than values reported for many traditional PS-based block copolymer electrolytes at room temperature.<sup>14–17</sup> The morphology of these BCP systems doped with IL has been inferred from microdomain characterization, while dynamic mechanical measurements showed a relatively high storage modulus, indicating that they are elastic solids.

## RESULTS AND DISCUSSION

The polyethylene-polar block copolymers were prepared according to reported procedures using a distorted diimine Pd<sup>II</sup> complex, showing living polymerization and yielding semicrystalline PE (38%) with low branching densities ( $\sim 17/1000$  carbon).<sup>18</sup> Metal-organic insertion light-initiated radical polymerization (MILRad) was employed to form PE-*b*-PMMA block copolymers for the ionic conductivity studies.<sup>18</sup> Block copolymer compositions were selected, which are envisioned to form a lamellar phase (Figure 1 and SI)



**Figure 1.** Polyethylene-*b*-poly(methyl methacrylate) synthesis

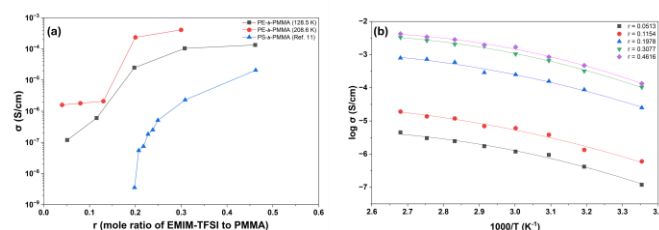
Polyethylene-block-poly(methyl methacrylate) (PE-*b*-PMMA) block copolymers with molecular weights of 128.5 kg/mol and 208.5 kg/mol and PE:PMMA composition of 45:55 in the block copolymer, as determined by <sup>1</sup>H NMR, were doped with EMIM-TFSI to prepare polymer electrolytes with *r* (the mole ratio of EMIM-TFSI to MMA) ranging from 0.04 to 0.5. Either toluene or mixed solvents of 1-chloronaphthalene (1-CN) and *N,N*-dimethylacetamide (DMAc) were used to prepare homogeneous casting

solutions. The solutions were then cast onto a bottom electrode of a blocking-electrode configuration in a glovebox under an inert nitrogen atmosphere at room temperature. The resulting films were further sequentially annealed under vacuum at 60 °C for 12 h and 100 °C for 48 h to remove residual solvent and minimize moisture uptake. Through-plane ionic conductivity was measured by electrochemical impedance spectroscopy using a Bio-Logic SP-150 electrochemical workstation. The ionic conductivity was calculated from

$$\sigma = \frac{L}{R_b A}$$

where  $L$  is the film thickness,  $R_b$  is the bulk resistance, and  $A$  is the electrode area. More details regarding the procedure are provided in the Supplementary Information (SI) document.

Figure 2(a) summarizes the room-temperature through-plane ionic conductivity of the PE-*b*-PMMA electrolyte series in comparison to similar BCP systems reported in the literature. The conductivity increase with respect to IL amount is very slight at low IL loading ( $r < 0.2$ ), however, it becomes sharp in the high IL loading regime, reaching the high  $10^{-4}$  S/cm range. It is difficult to attribute more than three orders of magnitude increase solely to the carrier-number effect of IL loading, rather than to a composition-dependent change in the effectiveness of ion-transport pathways within the microphase-separated film.

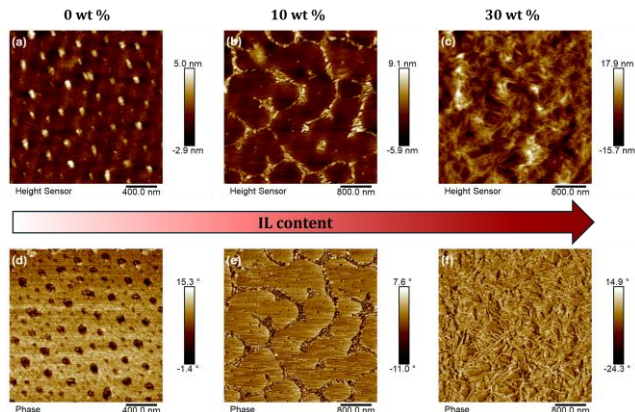


**Figure 2.** (a) Room-temperature through-plane ionic conductivity as a function of IL loading,  $r$ , for DMAc/1-CN-cast PE-*b*-PMMA ( $M_n = 128.5$  kg/mol) and toluene-cast PE-*b*-PMMA ( $M_n = 208.6$  kg/mol), with PS-*b*-PMMA/EMIM-TFSI literature data shown for comparison. (b) Temperature-dependent conductivity of the 128.5 kg/mol sample at different IL loadings, plotted as  $\log \sigma$  vs  $1000/T$ .

Comparing our dataset with the literature benchmark shown in Figure 2(a) reveals that there is an overlap point ( $0.1 < r < 0.2$ ) where, in that composition window, the ionic conductivity of similar BCP systems goes through a drastic increase, influenced by the molecular characteristics of the block copolymer. Prior studies on PMMA-containing block copolymers show that at low loading, the PMMA-rich conducting domains are still too sluggish for efficient long-range ion motion.<sup>11</sup> As EMIM-TFSI increases toward  $r \approx 0.2$ , the ionic liquid plasticizes PMMA and lowers its  $T_g$ , modulus, and relaxation time, affecting segmental motion positively.<sup>19</sup> At the same time, because the PE block excludes the ionic liquid, the local IL concentration inside the PMMA-rich phase increases more strongly than the overall composition

suggests, as this exact type of behavior was also reported in similar studies. Figure 2 (b) reinforces this interpretation as for all compositions,  $\log \sigma$  versus  $1000/T$  is clearly curved (fitting done to guide eye), indicating a Vogel-Tammann-Fulcher (VTF) behavior of transport. VTF-type behavior is common for polymer electrolytes as it accounts for the effect of polymer relaxation or segmental motion of polymer chains, unlike the simple Arrhenius model.<sup>20,21</sup>

The morphological evolution of PE-*b*-PMMA thin films with increasing IL loading using Atomic Force Microscopy (AFM) is summarized in Figure 3. In the neat copolymer (Figure 3 a,d), at this composition ( $f_{\text{PMMA}}=0.55$ ), lamellae is ex-

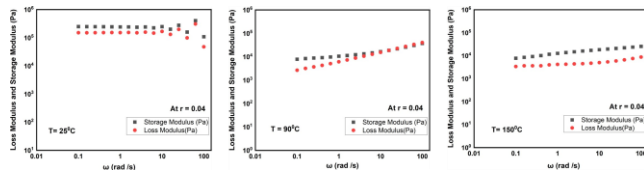


**Figure 3.** AFM height (a–c) and phase (d–f) images of PE-*b*-PMMA ( $f_{\text{PMMA}} = 0.55$ ) thin films with (a,d) 0, (b,e) 10, and (c,f) 30 wt% EMIM-TFSI. Scale bars and z-ranges as indicated.

pected morphology.<sup>22</sup> However, as can be seen from Figure 3 (d), roughly isotropic bright spots on the order of 60–100 nm in lateral dimension are distributed across a smooth matrix, making it inconsistent with the well-developed lamellar microstructure. As per our interpretation, PE crystallization kinetically traps the system before microphase separation can fully develop, producing isolated crystalline PE nodules embedded in a glassy PMMA-rich matrix. This is documented extensively for PE-containing block copolymers that BCPs with a high- $T_m$  block rarely show the lamellar morphology predicted by their  $f$  and  $\chi N$  because crystallization preempts microphase separation.<sup>23,24</sup> At 10 wt% IL (Figure 3 b,e), a polygonal network of IL-rich ridges surrounds flat PE/PMMA domains. The ridges reflect preferential partitioning of EMIM-TFSI into the PMMA block due to the well-established affinity between EMIM<sup>+</sup> cation and the carbonyl groups of PMMA.<sup>11,25</sup> Although this is consistent with the expected IL-in-PMMA selectivity, the IL-rich phase is still confined to isolated cell boundaries rather than forming a through-thickness network, which limits long-range ion transport at this loading. At 30 wt% IL (Figure 3c,f), the morphology transforms into a dense, percolating fibrillar network of PE lamellae embedded in a continuous, IL-swollen PMMA matrix. This indicates that, at this IL content, the PMMA block is sufficiently plasticized that its glass transition no longer arrests the system on the timescale of PE crystallization. This co-continuous architecture can be

thought of as a target microstructure for an SPE, with the IL-swollen PMMA providing continuous ionic pathways, while the percolating PE crystalline base giving the mechanical reinforcement and dimensional stability needed to maintain film integrity under stack pressure. The AFM data thus predict 30 wt% IL as the onset of the functional SPE regime, where ion-conducting and load-bearing phases are simultaneously continuous, which is in line with the ionic conductivity data displayed in Figure 2, where a sharp increase in conductivity is observed once the system reaches  $r = 0.2$  (30 wt% IL) regime.

Mechanical properties were evaluated by dynamic mechanical measurements (DMA) in the linear viscoelastic regime. The storage modulus was used to assess the solid-like character of the electrolytes. The frequency-dependent response of the  $r = 0.04$  electrolyte is summarized in Figure 4. At 25 °C, both moduli are essentially frequency-independent over nearly three decades of  $\omega$ . These parallel plateaus of loss and storage modulus, together with the storage modulus consistently exceeding the loss modulus, are characteristic of a soft elastic solid rather than a viscoelastic liquid,<sup>26</sup>



**Figure 4.** DMA shear rheology of PE-*b*-PMMA/EMIM-TFSI electrolyte with  $r = 0.04$  measured at 25, 90, and 150 °C.

which confirms that the film retains structural integrity at room temperature. At this low IL loading, the mechanical response is likely carried by the combined contribution of dispersed PE crystallites that act as physical cross-links, and the glassy, partially IL-plasticized PMMA matrix identified in the AFM images (Figure 3 b,e). Both moduli dropped by approximately an order of magnitude, approaching each other at high  $\omega$  after heating to 90 °C. We attribute this softening primarily to the  $\alpha$ -relaxation<sup>19,27</sup> of the IL-plasticized PMMA phase, whose effective  $T_g$  is depressed below that of neat PMMA by EMIM-TFSI. The PE crystallites remain intact (PE  $T_m$  typically  $\geq 110$  °C), so the network topology is preserved and  $G' > G''$  is mostly maintained across the accessible window. As temperature increases further (Figure 4, (c)), the  $\alpha$ -relaxation moves to higher frequencies and eventually exits the frequency window so we cannot see the dissipation peak associated with it and  $G''$  drops back below  $G'$ . What is fairly easy to interpret from this rheology data is that, even at an IL loading that does not yet produce a percolating crystalline scaffold, the PE-*b*-PMMA/EMIM-TFSI electrolyte behaves as a mechanically coherent elastic solid ( $G' > G''$ ) across the entire 25–150 °C window.

## CONCLUSION

In this letter, we have shown that solid polymer electrolytes made by adding different compositions of EMIM-TFSI into nearly symmetric PE-*b*-PMMA block copolymer produced

significantly high ionic conductivity ( $\sim 1$  mS/cm) at room temperature, which is considerably higher room temperature conductivity than comparable systems made with PMMA as an ion conducting block. In addition to having a low  $T_g$  structural block, we explain this significant enhancement in ionic conductivity in comparison to similar block copolymer systems with larger  $\chi$  (PE/PMMA) that leads to the sharper and narrower interfaces<sup>28</sup> and potentially less IL "leaking" into or near the structural block. Crystalline PE pins one end to a rigid lamella with essentially zero mobility, but the rest of the PMMA chain extends into a relatively unconstrained space where essentially no PMMA is trapped in constrained regions limiting mobility such as glassy polymer interfaces. In order to better understand the microstructure and conductivity relationship, AFM images of samples have been taken and analyzed with different IL compositions to observe morphological evolution of these electrolyte films, and been shown that these systems do not exhibit highly oriented block copolymer morphology expected from block composition, which can be explained by the high  $T_m$  of PE block, whose crystallization preempts microphase separation. However, with increased IL composition, morphology transforms into a dense, co-continuous network of PE lamellae embedded in a continuous, IL-swollen PMMA matrix. This morphological transition is paralleled by an increase in ionic conductivity across the 10–50 wt% IL range, and linear viscoelastic measurements confirm that the films retain solid-like response ( $G' > G''$ ) up to 150 °C, above the PE melting point. Overall, these polyethylene-block-polyacrylate electrolytes represent a promising route toward mechanically robust, thermally stable, and highly conductive solid-state electrolytes.

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## Author Contributions

The manuscript was written through the contributions of all authors.

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