# The Influence of Organic-Inorganic Composite Solid Electrolytes on Electrochemical Performance in Solid-State Lithium Batteries

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**Abstract.** Lithium batteries, as the most closely watched secondary batteries, exhibit outstanding performance. Nonetheless, the formation of lithium dendrites significantly compromises their safety and hampers further development. To mitigate the impact of lithium dendrites on batteries, solid-state lithium batteries have emerged, with solid electrolytes being of paramount importance. Traditional solid electrolytes are categorized into polymers, oxides, and sulfides. However, the inherent limitations of these materials impede their effectiveness. This study proposes a novel solid electrolyte consisting of organic and inorganic materials, aiming to harness the collective benefits of the two electrolyte types to improve the electrochemical performance of solid electrolytes. Empirical data from experiments on composite solid electrolytes, incorporating polyethylene oxide (PEO) with $Li_{6.4}La_3Zr_{1.4}Ta_{0.6}O_{12}$ (LLZTO) and $Li_{10}GeP_2S_{12}$ (LGPS), reveal that these organic-inorganic composite solid electrolytes exhibit high ionic conductivity, reduced interface impedance, and an extensive electrochemical window. It demonstrates that organic-inorganic composite solid electrolytes combining polymers with oxides or sulfides can integrate the advantages of both, achieving electrolyte materials with superior electrochemical performance. Finally, the existing problems are discussed, and future research trends are anticipated.

## 1 Introduction

Lithium batteries exhibit superior electrochemical performance. However, numerous incidents of battery explosions have drawn widespread societal concern regarding the safety of lithium batteries. The explosions of lithium batteries are primarily attributed to issues such as high temperature, high pressure, and the formation of lithium dendrites during multiple charging cycles. These problems can be addressed by improving electrode materials and electrolytes. However, these methods typically employ liquid electrolytes, which introduce problems related to leaks and thermal stability. In contrast, solid-state lithium batteries (SSLBs) utilize solid electrolytes instead of liquid ones, reducing the risk of electrolyte leakage and enhancing overall stability and safety. The electrolyte materials in SSLBs are diverse, including solid polymer electrolytes (SPEs), solid oxide electrolytes (SOEs), and solid sulfide electrolytes (SSEs). However, the reason SSLBs have not been produced on a large scale to date is due to different drawbacks of these three types of electrolytes: (i) SPEs have low conductivity at low temperatures and are at risk of ignition at high temperatures; (ii) high-performance SSEs are costly and pose process safety issues; (iii) there is difficulty in achieving good bonding between the grain boundaries in SOEs; (iv) rigid interfacial contact between solid electrolytes and solid electrodes leads to delayed reaction kinetics and "chemo-mechanical failures"[1]. Therefore, composite solid electrolytes (CSEs) have become a viable direction for advancing SSLB development, aiming to combine the advantages of polymer, sulfide, and oxide solid electrolytes to form an electrolyte system with high ionic conductivity (ICD), low cost, good processability, and cycling stability. This paper will focus on introducing organic-inorganic CSEs and outline the research progress of such electrolytes in recent years. It aims to elucidate the performance changes of solid-state batteries before and after using composite materials and to guide the direction for future research and development.

## 2 Inorganic solid electrolytes

### 2.1 Solid-state oxide electrolyte

In recent years, SOE have emerged as a focal point in the research of energy storage applications, attributed to their notable advantages including high safety performance, excellent thermal stability, cost-effectiveness, and environmental sustainability [2]. However, despite these benefits, SOE materials exhibit significant drawbacks, primarily their low ICD and the

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inherent hardness of the oxide grains [3]. The excessive hardness of these grains leads to the formation of interface voids and exacerbates interface resistance (IRS), undermining the electrolyte's efficiency. Introducing a liquid electrolyte to mitigate these issues results in the oxide being immersed in the liquid, thereby deviating from the concept of a fully solid-state battery. This approach essentially produces a semi-solid-state system or a solid-liquid hybrid solution. Such a compromise highlights the challenges in achieving the ideal all-solid-state battery configuration and underscores the need for continued research to overcome the limitations of SOE materials.

SOE is categorically divided into three primary structures: NASICON (sodium superionic conductor) type, garnet type, and perovskite type. The NASICON-type solid electrolytes are represented by the general formula $Li[A_2B_3O_{12}]$, encompassing variants such as $LiZr_2(PO_4)_3$ (LZP), $LiTi_2(PO_4)_3$ (LTP), and $LiGe_2(PO_4)_3$ (LGP) [2]. This structure is characterized by its straightforward synthesis, processability, excellent air and thermal stability, mechanical robustness, and high ICD. Nonetheless, the thermodynamic instability of internal $Ge^{4+}$ and $Ti^{4+}$ ions towards lithium, leading to their easy reduction by lithium, poses a significant challenge [2]. Garnet-type solid electrolytes, with the formula $Li_7La_3Zr_2O_{12}$ (LLZO), exhibit two crystallographic phases: cubic (c-LLZO) and tetragonal (t-LLZO) [2]. Owing to their enhanced stability and superior lithium-ion transport capabilities, garnet electrolytes have emerged as a focal point of research, demonstrating considerable potential. However, the issue of substantial IRS remains unresolved. Perovskite-structured solid electrolytes, denoted by the formula $Li_{3x}La_{2/3-x}TiO_3$ (LLTO), are noted for their high electrical conductivity, mechanical strength, and thermal stability. The primary limitation of this category is the high temperature required for their synthesis, which complicates the preparation process.

### 2.2 Solid-state sulfide electrolyte

Research on sulfide electrolytes can be traced back to the early 20th century. In comparison to oxide electrolytes, sulfide electrolytes exhibit superior ICD due to the larger ionic radius and lower electronegativity of $S^{2-}$ compared to $O^{2-}$. The weaker binding force between lithium ions and $O^{2-}$ results in a higher concentration of freely moving lithium ions, leading to an equivalent conductivity that rivals or even surpasses that of liquid electrolytes or even higher at low temperatures. Moreover, sulfide electrolytes possess benefits, including superior flexibility, straightforward fabrication procedures, outstanding mechanical characteristics, and optimal interface connectivity with electrode components. Sulphide electrolytes may be divided into three classes based on their crystalline state: glassy, glass-ceramic, and ceramic-crystalline electrolytes. The production of glass-ceramic sulphide electrolytes is primarily achieved by high-temperature pre-crystallization of glassy sulphide electrolytes. The latter are composed of an organic mixture of an amorphous and crystalline phase. Ceramic crystalline sulfide electrolyte materials perform exceptionally well overall, have high room-temperature ICDs, and have good lithium-ion transport routes. Prominent varieties of ceramic crystalline sulfide electrolytes include sulfur-lithium, sulfur-silver-germanite, and $Li_{10}GeP_2S_{12}$ types [4]. Based on composition, sulfide electrolytes can be categorized into binary and ternary sulfide electrolytes. Binary sulfide electrolytes are comprised of $Li_2S$ and $P_2S_5$, for example, $Li_3PS_4$ and $Li_7P_3S_{11}$, whereas ternary sulfide electrolytes are composed of $Li_2S$, $P_2S_5$, $ZS_2$ (Z = Si, Ge, Sn), such as $Li_{10}GeP_2S_{12}$ (LGPS) and $Li_6PS_5Y$ (Y = Cl, Br, I) [5].

Sulfide electrolytes are distinguished by their exceptional lithium ion ($Li^+$) conductivity at ambient temperatures. However, their high susceptibility to moisture absorption and vulnerability to atmospheric contaminants present significant challenges in their synthesis and storage. It is crucial to acknowledge that batteries incorporating sulfide electrolytes are prone to hydrogen sulfide ($H_2S$) gas emission during operation, which poses substantial expansion challenges and raises safety concerns for industrial applications. Although the introduction of additives has been explored as a strategy to reduce $H_2S$ gas formation, this method does not effectively resolve the fundamental issue. Furthermore, the interaction between sulfide electrolytes, specifically $Li_3PS_4$, and lithium metal may trigger a reduction reaction in the lithium metal. This reaction leads to the generation of products such as $Li_3P$ and $Li_2S$, which are characterized by their low ICD, further complicating the application of sulfide electrolytes in battery technology. This process causes increased interface impedance, ultimately forming a stable solid electrolyte membrane (SEM) layer that contributes to IRS. Consequently, this instability affects the interaction between the SSE and both lithium metal and traditional oxide cathode materials. For instance, the reaction between $Li_{10}GeP_2S_{12}$ and lithium metal results in an SEM layer containing $Li_3P$, $Li_2S$, and Ge-Li alloy, which facilitates electronic transmission. This phenomenon contributes to the thickening of the SEM layer and an increase in interface impedance. Additionally, it should be noted that the electrochemical stability window (ESW) of sulfide electrolytes is quite narrow [4].

## 3 Solid-state polymer electrolyte

Polymer solid electrolytes are primarily composed of lithium salts and a polymer matrix. In this structure, the polymer matrix acts as the medium for lithium-ion transport, while the lithium salts serve as charge carriers. Ideally, the chosen lithium salt should possess a lower lattice energy to facilitate its dissociation; simultaneously, the polymer matrix should have a higher dielectric constant, which aids in the dissociation and movement of ions [6]. Among the many commonly used polymer matrix materials are PEO and its derivatives, polyacrylonitrile (PAN), and polyvinylidene fluoride (PVDF).

### 3.1 Polyethylene oxide matrix

The PEO matrix has become one of the most researched solid electrolyte matrices due to its strong ability to dissociate lithium salts, excellent electrochemical stability, low glass transition temperature, and good flexibility [7]. The chemical structure of PEO is H-(O-$CH_2$-$CH_2$)$_n$-OH, typically with a molecular weight exceeding 20,000 g/mol. Its unique chemical molecular structure (O-$CH_2$-$CH_2$)$_n$ can form complexes with many lithium salts (such as LiTFSI), promoting $Li^+$ transport through segmental motion, and making it a good lithium-ion conductor [7]. At room temperature, the PEO matrix consists of amorphous and crystalline regions. In the amorphous regions, $Li^+$ is transported through the movement of polymer chains, while in the crystalline regions, the segmental motion of ions slows down, which is unfavorable for ion migration. Therefore, the mainstream view is that ion conduction primarily relies on the amorphous regions. Within the temperature range below the glass transition temperature, the polymer is mainly in a crystalline state, and the lower the glass transition temperature, the more flexible the polymer chains [7]. Hence, to achieve high ICD and high mechanical strength of PEO at room temperature, measures such as reducing the crystallinity and glass transition temperature of the polymer are generally adopted to accelerate the movement of polymer segments [7].

### 3.2 Disadvantages of polyethylene oxide matrix

Research has shown that PEO electrolytes have a high degree of crystallinity at room temperature, ranging between 70% and 85% [6]. Due to the slow segmental motion of $Li^+$ in the crystalline regions, this results in low conductivity of single PEO electrolytes at room temperature. Additionally, the hydroxyl groups (-OH) are easily oxidized at high voltages, leading to a low ESW for PEO electrolytes, which makes it challenging to withstand voltages higher than 4V. This significantly limits the energy density of the batteries.

## 4 Organic-inorganic composite solid electrolyte

### 4.1 Polymer/oxide composite solid-state electrolyte

Given that semi-solid electrolytes have not effectively resolved the issue of lithium dendrite formation, posing persistent risks of fire and explosions alongside other safety concerns, the development of a ceramic-in-polymer and polymer-in-ceramic (CIP-PIC) structure for CSEs has been proposed. This innovative manufacturing technology aims to address the challenge of IRS attributed to interfacial voids, thereby enhancing the performance of SSLBs. Polymer electrolytes are categorized into gel polymer electrolytes (GPE) and SPE, distinguished by their composition. The primary distinction lies in the presence of a liquid plasticizer in GPEs, whereas SPEs are devoid of such additives. This differentiation is crucial for understanding their respective behaviors and applications in battery technology.

The garnet-structured solid electrolyte, LLZO, has emerged as a significant area of research in recent years. In this context, Chen et al. developed an innovative, flexible composite solid polymer electrolyte (CSPE) membrane. This was achieved by incorporating LLZTO ceramic particles into a PVDF-HFP/LiTFSI-based SPE through a hot-pressing technique that obviates the need for organic solvents [8]. Similarly, Wang et al. fabricated Ga-LLZO nanofibers using the electrospinning method and integrated them into a PVDF-HFP polymer to create a composite electrolyte [9]. Furthermore, Yu et al. introduced LLZTO ceramic nanoparticles into a PEGDA polymer matrix via a tape casting and UV curing process, leading to the design of a novel composite electrolyte [10].

These developments underscore the versatility of LLZTO ceramics in combination with various polymers such as PEO, PVDF, and PEGDA, among others, to engineer solid electrolytes. For instance, Huang et al. crafted a highly ionic conductive CSE film by merging 1,3-dioxolane (DOL) with the highly ionic conductive $Li_{1.5}Al_{0.5}Ge_{1.5}(PO_4)_3$ (LAGP) ceramic filler [11]. These advancements highlight the potential of polymer-oxide CSEs to achieve effective bonding at the grain interface, indicating substantial prospects for development in this field.

### 4.2 Polymer/sulfide composite solid-state electrolyte

Sulfide-based electrolytes are renowned for their high ICD, robust mechanical properties, and excellent compatibility with sulfur cathodes. Despite these advantages, their electrochemical performance, especially in terms of actual energy density, falls markedly short of theoretical expectations. This discrepancy is attributed to challenges such as the high-pressure compression required for inorganic solid electrolyte particles and the 'chemomechanical' issues that arise. To address these challenges and enhance the performance of sulfur cathodes in SSLSBs, various strategies have been employed. A prominent method involves the creation of composite sulfur cathodes by integrating active materials with solid electrolytes. However, while this method may improve material contact, it has shown limited success in boosting cycling performance and could inadvertently escalate production costs.

Meirong Li introduced a novel SSLSB structure utilizing an LGPS/PEO composite electrolyte and a sulfurized polyacrylonitrile (S/PAN) cathode, resulting in enhanced wettability and reduced charge transfer resistance. The composite material shows significantly improved cycling performance, with the S/PAN battery demonstrating superior initial capacity and maintaining a capacity of 588 mAh $g^{-1}$ after 50 cycles, compared to the

rapid decay observed in traditional S/C cathode batteries [12].

The LGPS electrolyte exhibits high lithium-ion conductivity, however, its practical application is hindered by the instability of the lithium metal anode. Therefore, the PEO/LGPS composite can be seen as an excellent example of polymer/ceramic composite electrolytes. The process of producing the PEO/LGPS composite material is depicted in Figure 1. PEO and LiTFSI were combined with varying amounts of LGPS in acetonitrile at a molar ratio of 10:1. Following mechanical stirring for 12 hours, the mixture was coated onto a poly (ethylene terephthalate) foil to create an electrolyte film. Ceramic powders were evenly dispersed within the polymer matrix, and composite materials with ceramic content of 10%, 30% and 50% (referred to as LGPS10, LGPS30, and LGPS50) were chosen as experimental variables [13].

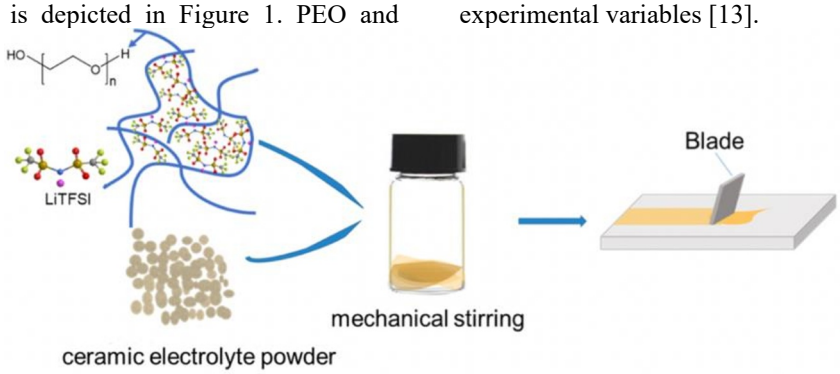


**Fig. 1.** Manufacturing process of PEO/LGPS composite electrolyte [13].

## 5 Electrochemical performances

### 5.1 Ionic conductivity

#### *5.1.1 Ionic conductivity of oxide-polymer composite solid-state batteries*

The relationship between (100-x) wt% [PEO-LiTFSI] - x wt% LLZTO ionic conductivity and temperature can be obtained from Figure 2. At all temperatures, the ionic conductivity of 90 wt% [PEO-LiTFSI] – 10 wt% LLZTO and 80 wt% [PEO-LiTFS] - 20wt% LLZTO is always higher than that of PEO-LiTFSI. According to the research data of Chen et al., 90 wt% [PEO-LiTFSI] – 10 wt% LLZTO reaches $1.17\times10^{-4}$ S $cm^{-1}$ at 30℃ and $1.58\times10^{-3}$ S $cm^{-1}$ at 80 ℃. When the temperature is 55 ℃, the ionic conductivity reaches more than $10^{-4}$ S $cm^{-1}$, illustrating that the combination of oxide and polymer can effectively improve the ionic conductivity of electrolytes [8]. Besides, Figure. 2b also shows that ionic conductivity decreases with the increase of LLZTO content when LLZTO content is greater than or equal to 10 wt%. When LLZTO content is more than 20 wt%, the ionic conductivity is less than that of the PEO-LiTFSI battery. This shows that the appropriate addition of LLZTO ceramic particles can improve the ionic conductivity of lithium batteries.

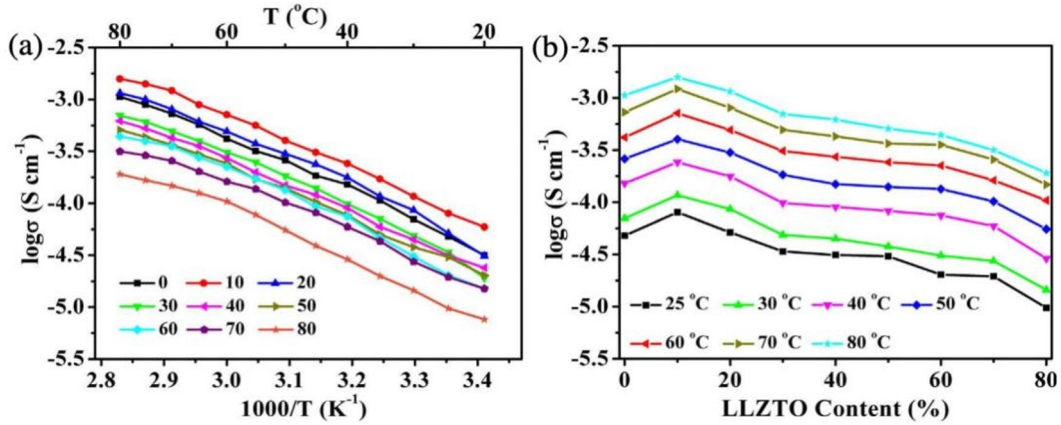


**Fig. 2.** (a) The variation of ionic conductivities of [PEO-LiTFSI] - LLZTO with temperature (b) The change of ionic conductivities of [PEO-LiTFSI] - LLZTO with LLZTO content [8].

#### *5.1.2 Ionic conductivity of sulfide-polymer composite solid-state batteries*

Recent research focuses on the critical role of LGPS and LiTFSI components in enhancing ion conduction at the interface of an LGPS (PEO-LiTFSI) hybrid material, with an emphasis on determining the optimal content ratio for improved lithium-ion mobility. Through EIS, it was found that the overall conductivity of the hybrid electrolyte film increases with LGPS content, reaching its peak conductivity at a composition of 70 wt% LGPS,

as depicted in Figure. 3a. This optimal conductivity is further enhanced with an increase in LiTFSI content, achieving maximum efficiency at an EO:LiTFSI ratio of 9:1, as demonstrated in Figure. 3b. At this composition, Fig. 3c shows that the ion conductivity of the 70 wt% LGPS (9:1) (PEO-LiTFSI) hybrid is 0.22 mS $cm^{-1}$, which not only exceeds that of the standard PEO-LiTFSI but also presents significantly lower activation energy for lithium-ion transport compared to both individual components. This indicates a synergistic effect within the hybrid material, facilitating superior ion conduction.

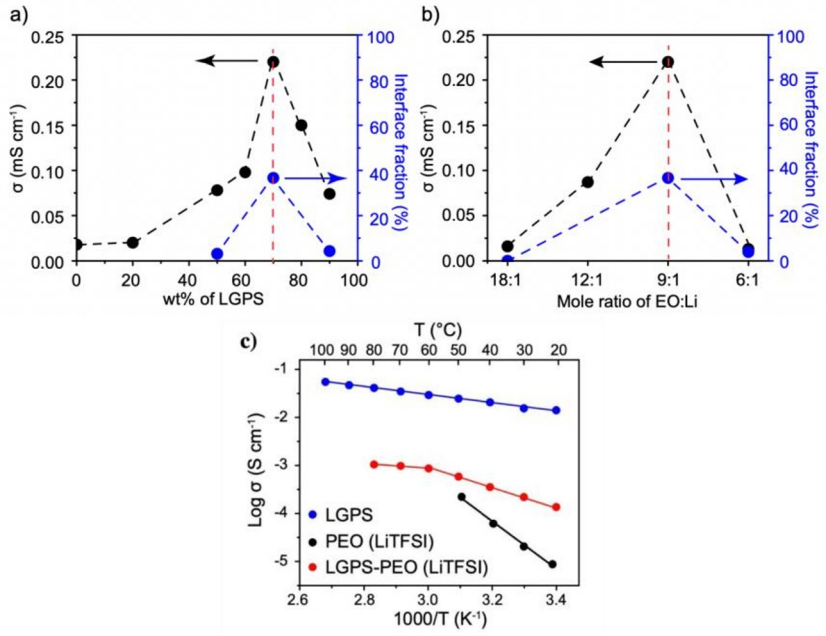


**Fig. 3.** (a) The function of wt% of LGPS on the interface fraction and conductivity of LGPS (PEO-LiTFSI) with the mole ratio of EO to LiTFSI of 9:1 at 22. (b) The mole ratio of EO to LiTFSI on the conductivity and interface fraction of LGPS (PEO-LiTFSI) with a wt% of 70 at 22. (c) The plots of conductivity of different electrolytes [14].

## 5.2 Interface resistance

### *5.2.1 Interface resistance of oxide-polymer composite solid-state batteries*

As depicted in Figure 4a, the impedance spectrum of the electrolyte was recorded under a constant direct current (DC) bias voltage of 4.8V at 55 ℃. During this charge-discharge cycle, the impedance and IRS of the SS|$PEO_8$-LiTFSI|Li cell were observed to increase progressively over time. Conversely, as illustrated in Figure 4b, the IRS of the SS|$PEO_8$-LiTFSI-10 wt% LLZTO|Li cell exhibited only minor variations within a 72-hour period. This observation suggests that incorporating LLZTO significantly diminishes both the impedance and IRS in lithium batteries.

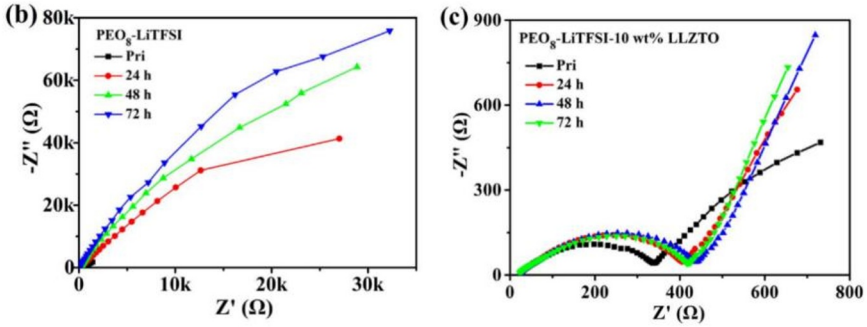


**Fig. 4.** The impedance spectra of (a) SS|$PEO_8$-LiTFSI|Li battery; (b) SS|$PEO_8$-LiTFSI-10 wt% LLZTO|Li battery [8].

### *5.2.2 Interface resistance of sulfide-polymer composite solid-state batteries*

Figure 5 displays the results of EIS conducted on composite electrolytes (LGPS and PEO) in symmetric stainless-steel batteries at a temperature of 20 ℃. The surface structure of the film changes as the ceramic content varies. Experimental variables were chosen as composite materials with LGPS ceramic contents (wt%) of 10%, 50%, and 90%, denoted as LGPS10, LGPS50, and LGPS90, respectively. It is evident from the data that the impedance of LGPS10 is significantly higher than that of LGPS50 and LGPS90. This suggests that the impedance of the LGPS/PEO composite is influenced by the LGPS content, with lower LGPS content resulting in lower impedance.

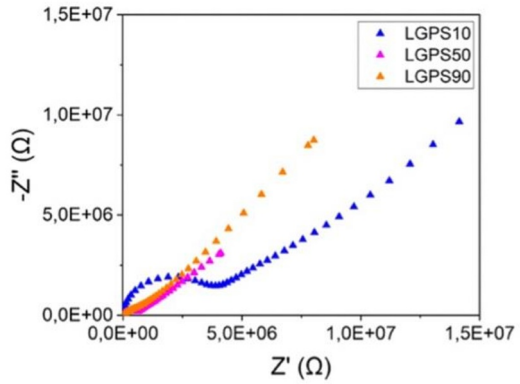


**Fig. 5.** EIS spectra of the three composite electrolytes at 20 ℃ [13].

### 5.3 Electrochemical stability window (ESW)

#### *5.3.1 ESW of oxide-polymer composite SSBs*

Figure 6 shows that the addition of 10 wt% LLZTO significantly increases the ESW. The $PEO_8$-LiTFSI membrane has an ESW of about 4.3V, while the composite electrolyte containing LLZTO remains stable at 5.0V [8].

Wan et al. showed that the addition of LLZO nanowires and LLZO particles to PL and PLLM can expand the ESW from 5.3 to 5.5 and 6.0V, while the PLLM and PL electrolytes become unstable from 3.5 V, indicating that the composite electrode has high stability for lithium metal [15].

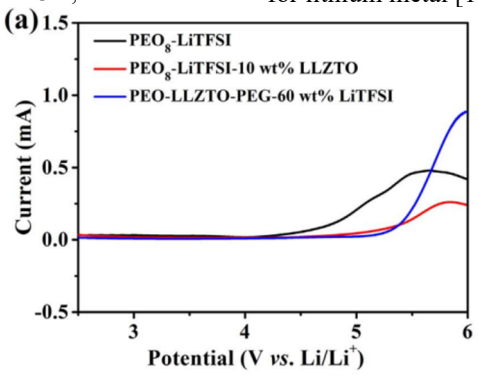


**Fig. 6.** The ESW of PEO8-LiTFSI-10wt% LLZTO and PEO-LLZTO-PEG-60wt% LiTFSI samples at 55 °C; scan rate of 1 mV $s^{-1}$ [8].

#### *5.3.2 ESW of sulfide-polymer composite SSBs*

According to Figure 7, PEO-LiTFSI has an ESW of about 4.9 V at 40 ℃. After being compounded with 1% LGPS, its ESW increases by nearly 0.6 V, which is undoubtedly a great improvement. Compared with high-cost and complex formulations, adding small amounts of LGPS into PEO-LiTFSI can greatly broaden the ESW of $PEO_{18}$-LiTFSI, and cost performance is higher. This is related to the presence of SH groups on the surface of LGPS inorganic particles, which are very conducive to promoting the combination of TFSI- and LGPS, leading to a decrease in the mobility of anions [16].

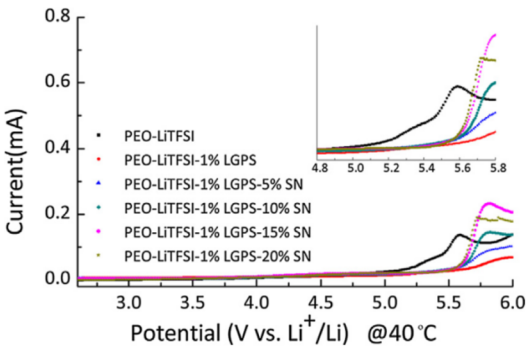


**Fig. 7.** The LSV curves of PEO-LiTFSI and LGPS-PEO(LiTFSI) on the electrochemical stability window at 40 with a scan rate of 1 mV $s^{-1}$ [16].

## 5 Conclusion

SSLBs, with their excellent energy density and safety characteristics, demonstrate vast potential for widespread applications. In such battery systems, high-performance solid electrolytes play a crucial role. By adding inorganic ceramic fillers (such as oxides or sulfides) to an organic polymer matrix, it is possible to effectively reduce the crystallinity of the polymer, thereby producing a CSE with high ICD, a wide ESW, and good mechanical properties and interface compatibility. Although significant achievements have been made in research, CSEs still face major challenges; addressing these issues is a key direction for future research:

Firstly, regarding the transport mechanism of lithium ions in CSEs, existing research has not yet delved deeply, lacking a universal theory that can guide the design and preparation of electrolytes. Therefore, a deeper exploration of the lithium-ion transport mechanism is needed. Further, although the room temperature ICD of CSEs has been improved compared to pure organic electrolytes, there is still a gap compared to pure inorganic solid electrolytes, necessitating further enhancement of their room temperature ICD. Finally, interface issues remain a significant challenge for composite electrolytes. Although improvements have been made in the solid-solid interface with electrodes compared to inorganic electrolytes, problems such as lithium dendrites, electrode/electrolyte interface reactions, and the complex multiphase interfaces within the composite electrolyte still require further research to address these challenges.

## Authors contribution

All the authors contributed equally and their names were listed in alphabetical order.

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