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Interface Failure Mechanisms and Stabilization Strategies in Sulfide-Based All-Solid-State Lithium Batteries

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Abstract. Sulfide-based all-solid-state lithium-ion batteries, characterized by their lithium metal anodes and elevated energy density prospects, are viewed as a highly promising architecture for high-performance all-solid-state power cells. Nevertheless, within actual full-cell setups, the interface stability continues to encounter significant hurdles including chemical reactivity at the high-voltage cathode side and lithium-metal/sulfide-electrolyte boundary interactions. Low-pressure operation requires simultaneous control of chemical side reactions, mechanical contact, and uniformity of lithium deposition. Therefore, this paper systematically reviews the interface failure mechanisms of sulfide all-solid-state lithium batteries, and summarizes the research progress of interface stabilization strategies in recent years from several aspects, providing a reference for the design of interfaces in sulfide all-solid-state batteries.

Keywords: All-solid-state battery, Interface Failure Mechanisms, Sulfide solid electrolytes

1. Introduction

Currently, commercial liquid lithium-ion batteries (LIBs) have been widely used in electric vehicles and portable electronic devices [1]. However, the traditional organic liquid electrolyte and graphite anode system are approaching the physical limit in terms of energy density. If the energy density is further enhanced by introducing high-nickel positive electrodes or silicon-based negative electrodes, the electrochemical compatibility of the liquid electrolyte will deteriorate sharply [2, 3]. These safety hazards and performance bottlenecks have restricted the further development of liquid lithium batteries. All-solid-state lithium-ion batteries (ASSLBs) substitute the combustible liquid electrolyte and porous separator for a solid electrolyte, providing enhanced safety benefits for the cell while maintaining compatibility with high-voltage cathodes and high-capacity metal lithium anodes. Consequently, they are viewed as the primary technology for the future development of high-energy-density energy storage [4, 5].

The sulfide solid-state electrolyte possesses extremely high room-temperature ionic conductivity (10^{-3}~10^{-2} S/cm), low grain boundary impedance, and excellent mechanical ductility. Its unique texture enables it to easily form a tight interface contact with the electrode, demonstrating favourable processability, although interfacial compatibility with lithium metal remains challenging. Therefore, it has become the most promising technical path for achieving large-scale application of high energy density all-solid-state batteries [6]. At present, the solid-state electrolytes based on

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sulfides mainly include three types: lithium argyrodite (LPSX), lithium germanium phosphorus sulfur (LGPS), and Li_2S-P_2S_5 type [7]. Although the sulfide electrolyte membrane exhibits certain lithium dendrite suppression ability under ideal conditions due to its low surface porosity and high Young's modulus, in the actual all-cell system, the inherent defects at the solid-solid interface become the core bottleneck restricting its commercialization. These problems are coupled with each other, jointly leading to a continuous increase in interface impedance, rapid capacity decline and short-circuit risk.

Regarding this bottleneck, this article will conduct an in-depth analysis from three aspects on the failure mechanism of the interface chemistry evolution in sulfide solid-state batteries, and several stabilization strategies were proposed specifically. The feasible modification strategies and research progress were elaborated in detail, which have significant academic value and technical significance for promoting the commercial application of all-solid-state batteries.

2. Interfacial failure mechanisms in sulfide-based all-solid-state batteries

The solid-solid interface, which serves as the main site for lithium-ion charge transfer and electrochemical reactions, is the most vulnerable and crucial part in the battery system, and its failure mechanism is also more complex [8]. The root cause lies in the inherent incompatibility between the electrode and the solid electrolyte in terms of chemical properties, electrochemical window, and mechanical behavior. This is mainly manifested as interface physical contact failure, interface side reactions, space charge layer effect, and lithium dendrite growth, among other bottleneck issues [9, 10]. This section will systematically analyze the above-mentioned interface failure mechanisms, with the aim of providing a theoretical basis for interface optimization and the design of low compaction strategies.

2.1. Electrochemical and chemical instability at the solid-solid interface

The inherent chemical and electrochemical stability window of sulfide electrolytes is relatively narrow, with high instability and insufficient chemical compatibility with mainstream positive and negative electrode materials. It is prone to thermodynamically-driven spontaneous chemical reactions and electrochemical redox decomposition. When sulfide solid-state electrolytes come into direct contact with oxide cathodes, the intrinsic thermodynamics are unstable. As a result, an interface chemical reaction occurs spontaneously between the two, generating transition metal sulfides such as Co_xS_γ and Ni_xS_γ as by-products. These low-ion-conductivity by-products will accumulate at the interface and form a high-resistance layer, which is the main reason for the initial high interface impedance of the battery in terms of chemical stability [11]. Correspondingly, the interface chemical instability on the negative electrode side is also prominent: The highly reactive metallic lithium is extremely prone to undergo reduction reactions with the S2- and P3- groups in the sulfide electrolyte, generating solid-state electrolyte interface layers (SEI) composed of components such as Li2S and Li3P. The ionic conductivity of this interface layer is relatively low, which severely hinders the rapid transmission of lithium ions across the interface. The low-conductivity products further aggravate the deterioration of the interface [12].

Regarding electrochemical durability, the core issue resides in the reality that electrolytes possess a restricted electrochemical range of stability, and their fundamental highest occupied molecular orbital (HOMO) plus lowest unoccupied molecular orbital (LUMO) electronic energy structural arrangement fails to establish a thermodynamic alignment with the Fermi levels (μc plus μa) of the cathode and anode materials within the cell. Based upon the energy level matching concept, the

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highest occupied molecular orbital (HOMO) energy position of the electrolyte needs to be below the Fermi level μc of the cathode to guarantee that the electrolyte does not suffer from oxidation breakdown at elevated potentials; its lowest unoccupied molecular orbital (LUMO) energy position must be above the Fermi level μa of the anode to avoid the electrolyte from being reduced at depressed potentials. This energy level matching relationship forms the intrinsic electrochemical stability window (ESW) of the electrolyte. Once the energy levels mismatch, a series of irreversible interfacial side reactions will occur, seriously damaging the performance of the battery [13, 14].

As shown in Figure 1 [13], it is a schematic diagram illustrating the ion transfer between lithium metal and the solid-state electrolyte. Wenzel et al. conducted a quantitative study on the interface reaction between lithium lanthanum titanium oxide (LLTO) solid electrolyte and lithium metal by combining in-situ X-ray photoelectron spectroscopy with electrical characterization [13]. They confirmed the mixed conductive intermediate phase (MCI) type interface chemical failure mechanism. After 1000 minutes of lithium deposition, the proportion of Ti^{4+} at the LLTO interface dropped below 50%, while 25% of metallic Ti^0 was generated; the Ti element was gradually reduced to Ti^{3+}, Ti^{2+} until it reached the elemental state, directly proving that the electrolyte underwent deep reduction decomposition. After the lithiumization reaction, the electronic conductivity of the electrolyte increased from the intrinsic value of 1.32×10^{-11} S/cm to 0.12 S/cm, an increase of nearly 10 orders of magnitude; the interface transformed from a pure ionic conductor to an ionic-electron mixed conductor, losing the electron insulating barrier [13].

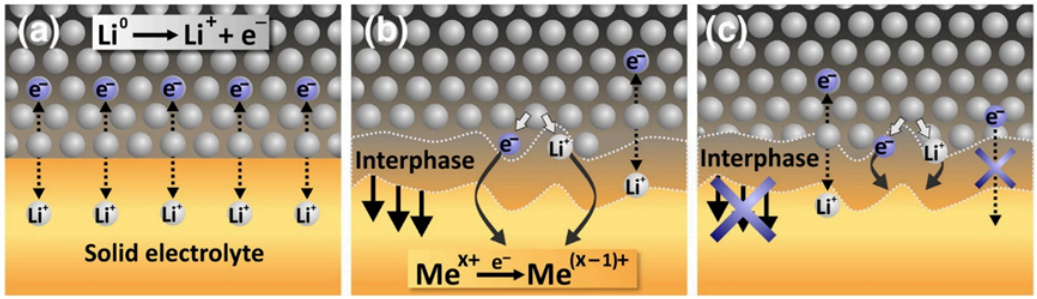


Figure 1. Ion transport between lithium metal and solid electrolyte [13]

2.2. Mechanical failure

Throughout the charge-discharge cycles of solid-state batteries, substantial electrode volumetric stress occurs: the relative dimensional shift ratio for the lithium metal anode during plating/exfoliation is nearly 100%, while the transition metal oxide cathode likewise experiences 2% to 5% structural swelling/shrinking throughout the lithium extraction/intercalation procedure. Although the sulfide electrolyte has a certain degree of ductility, it still cannot adapt to volume changes through flowing and infiltration like the liquid electrolyte, and is prone to mechanical contact failure.

The sulfide electrolyte is limited by the surface micro-roughness and porosity, resulting in a relatively small initial effective solid-solid contact area. During the cycling process, the strong electrochemical-mechanical coupling effect caused by the repeated lithium extraction and insertion of the electrode material will lead to significant stress concentration within the active particles and at the interfaces, thereby triggering the initiation and propagation of microcracks. This chemical-mechanical degradation not only causes the electrode particles to flake off and lose their physical contact with the electrolyte, but also alters the chemical potential of the local lithium, reduces the charge transfer kinetics, and ultimately leads to the interruption of the interface conductive pathway

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and the rapid decay of reversible capacity (Figure 2) [14-17]. Liu et al. [18] used in-situ transmission electron microscopy to conduct real-time observations on the lithiumization process of single-crystal silicon nanowires along the <112> crystal direction. They directly confirmed the highly anisotropic expansion characteristics of silicon lithiumization and the fracture mechanism driven by internal stress: anisotropic lithiation-induced strain can initiate and propagate cracks.

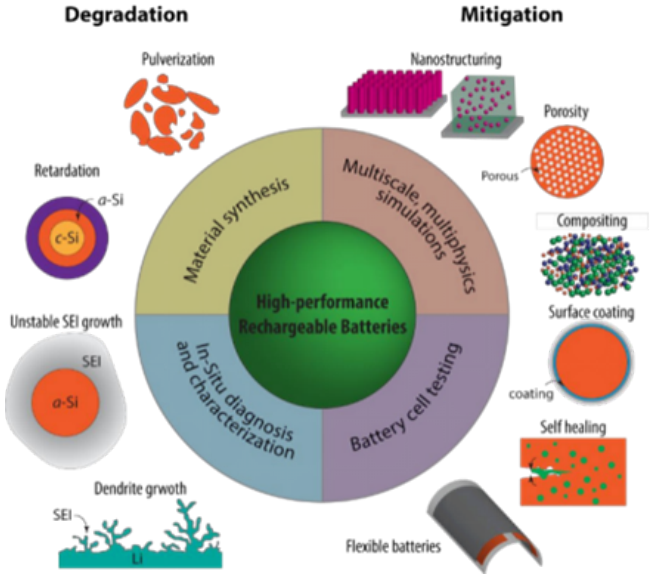


Figure 2. Advanced characterization techniques, degradation mechanisms, and mitigating strategies of high-volume-change anode materials [14]

2.3. Lithium dendrite growth mechanism in solid electrolytes

The development of lithium spines represents the primary obstacle hindering the practical implementation of all-solid-state lithium metal batteries utilizing sulfide-based electrolytes. This phenomenon not only triggers the irreversible depletion and capacity loss of active lithium, but also increases the risk of dendritic penetration into the solid electrolyte interface, resulting in internal shorts and presenting significant safety risks [15, 19]. On the one hand, within the solid electrolyte matrix, there are numerous microscopic defects, such as grain boundaries, pores, cracks or surface scratches, along with a large number of microscopic voids and contact-inadequate areas. These defects occur because the local ion conductivity is higher or the mechanical strength is weaker. As a result, lithium ions can only deposit at a limited number of effective contact sites. When the local current density exceeds the Critical Current for Stripping (CCS), lithium metal will preferentially form moss-like deposits at these locations, creating a "tip effect", which accelerates the deposition process and promotes the formation of dendritic nuclei. On the other hand, the spontaneously formed solid electrolyte interphase layer (SEI) at the interface has an uneven impedance distribution, which severely hinders lithium ion conduction. At the same time, lithium ions preferentially deposit in areas with lower impedance, eventually forming the nucleation points for lithium dendrites [20]. Figure 3 show the diagrams of contact loss and the growth mechanism of lithium dendrites at the interface [21].

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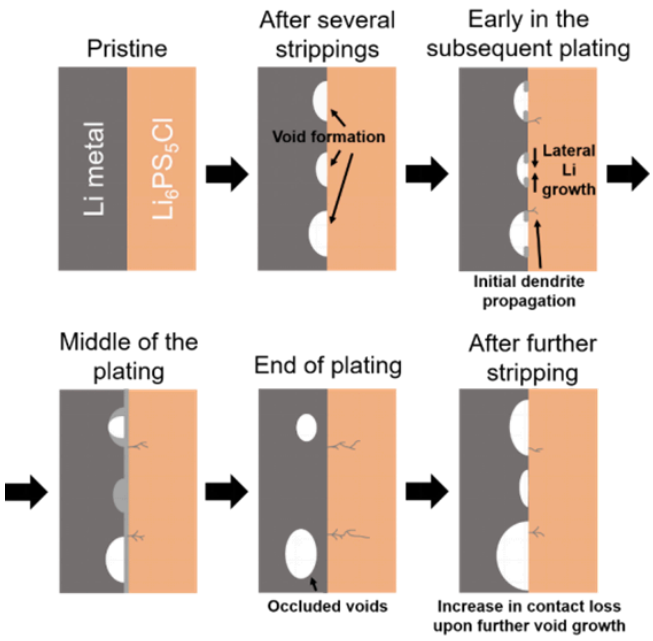


Figure 3. Lithium dendrite growth at the interface [21]

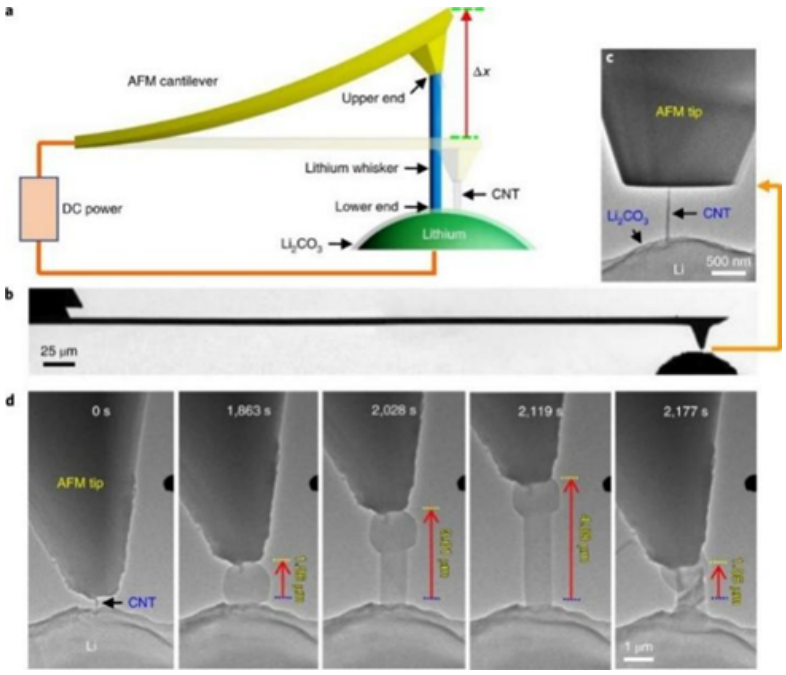


Figure 4. Stresses generated during the growth of lithium dendrites [19]

As shown in Figure 4, the tremendous mechanical stress generated during the lithium deposition process is an important contributor for the dendrite growth across the medium [19]. When lithium ions continuously deposit as in-situ metallic lithium at the grain boundaries or within the pores, the volume expansion of lithium metal will generate extremely high local stress at the crack tips. When this stress exceeds the mechanical strength limit of the sulfide solid electrolyte, it will cause the material to fracture or undergo creep. The cracks will then propagate forward, and the newly formed crack voids will be further filled by lithium, thereby forming a cycle of "lithium deposition → stress

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accumulation → crack expansion → new deposition space". In this way, the lithium dendrites, along with the cracks, extend into the solid electrolyte, eventually penetrating the entire electrolyte layer, and ultimately causing a short circuit within the battery [15].

3. Stabilization strategies for sulfide all-solid-state lithium batteries

To maintain the close contact of the solid-solid interface, high external stacking pressure (100 - 600 MPa) is usually applied during the laboratory stage [21]. However, operating under high external pressure increases the complexity of module design and the cost of overlap, and it is highly likely to cause irreversible mechanical damage to the battery cells [22]. Developing a low-pressured operation strategy that can ensure the long-term stability of battery performance has become a key point for industrialization. Regarding the problems of interface contact peeling and dendrite accelerated penetration under low stacking pressure, current research mainly focuses on three aspects: intrinsic modification at the material level, interface engineering adaptation, and design at the structural/systemic level.

3.1. Material-level modification

Material-level modification involves optimizing the intrinsic physical and mechanical properties of the electrolyte and electrode materials through methods such as element doping, anion substitution, or composite polymer/sulfide incorporation, to enhance the electrochemical stability and mechanical strength of the sulfide electrolyte. Some studies have also explored the introduction of interface buffer components into the sulfide electrolyte to improve compatibility with the cathode [23].

Among various methods, one common approach is to select or modify the cathode materials with low volume change rate during lithium extraction to reduce the lattice expansion and contraction during the charging and discharging processes. Qiang et al. [24] proposed introducing In_2O_3 on the surface of high-nickel NCM90 cathodes to locally convert the residual lithium into a $LiInO_2$ & In_2O_3 epitaxial protective layer. This approach not only prevented volume expansion but also mitigated interface side reactions. Moreover, it demonstrated excellent cycling stability even at a high load of 9 mg/cm².

Among the strategies for regulating the mechanical and transport properties of sulfide electrolytes, elemental substitution is a widely investigated approach for enhancing the stability of the Li interface. By inducing lattice disorder in chalcogenide electrolytes (such as $L_{i6}PS_5I$, Na_3PS_4) through non-equivalent/equivalent doping (such as V^{5+}, Ge^{4+}, Si^{4+}) or anion substitution (such as Se replacing S), the ion diffusion channels are expanded and the migration energy barrier is reduced [25]. Zhao et al. [26] introduced a dual-component Al/Zr addition approach to improve the single-phase NCM811 host. The volumetric contraction ratio of NCM811 during 4.4V operation was decreased by roughly 25.5%. In the integrated cathode following cycling (Figure 5), the pore volume of this substance was notably smaller than that of SC811. Under a modest compaction force of 2 MPa, this electrode still attains a specific discharge capacity of 169.5 mAh g^{-1} (at 0.2C), and the capacity stability ratio after 50 cycles is 90.8% [26].

Designing and introducing amorphous sulfide fillers to improve the interface is also a very effective strategy. Lai et al. [27] constructed a composite solid-state electrolyte by introducing amorphous $3Li_2S$-$2P_2S_5$ sulfide fillers into the PVDF-LiTFSI polymer electrolyte, which simultaneously possessed high ionic conductivity and interface self-stabilization ability. This electrolyte can generate an in-situ Li_2S modified interface layer on the lithium negative electrode

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during the cycling process, inhibiting the de-fluorination decomposition side reaction of PVDF, and significantly enhancing the long-term cycling stability of the symmetric battery and the all-cell.

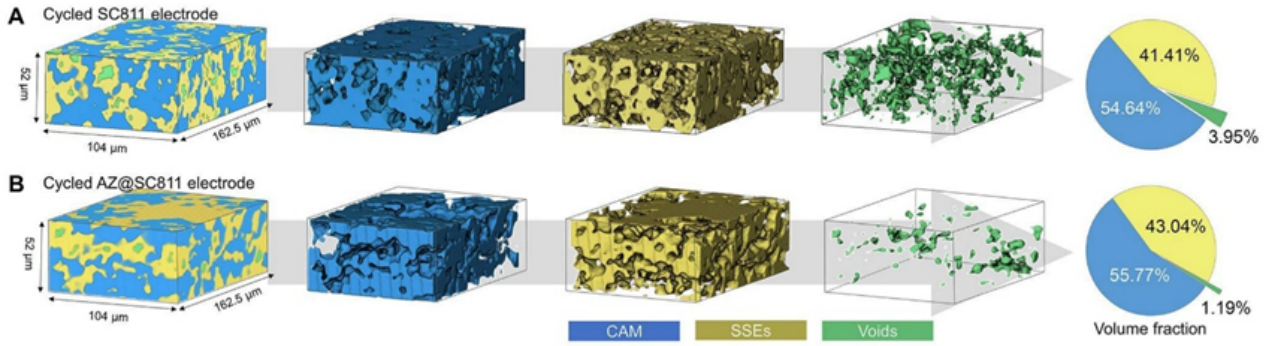


Figure 5. Synchrotron X-ray CT reconstruction with volume rendering reveals the 3D architectures and volume fractions of SC811 and AZ@SC811 composite cathodes after electrochemical cycling [26]

3.2. Interface engineering optimization

Interface engineering, also known as active material surface coating modification, involves constructing an artificial interface layer (such as oxide, fluoride, phosphate coatings, etc.) on the surface of high-voltage positive electrode particles or regulating the interface chemical reactions. Introducing a functionalized intermediate layer (SEI) at the electrode-electrolyte interface can compensate for the interface contact defects under low pressure, prevent interface side reactions, and reduce interface impedance. Meanwhile, some ionic conductive coatings can also facilitate the migration of lithium ions at the interface, thereby improving the kinetic performance [28].

The highly reactive lithium metal anode is prone to intense interfacial side reactions with the solid-state sulfide electrolyte, which severely limits the cycle stability and service life of all-solid-state batteries. Therefore, the in-situ construction of a multi-component synergistic hybrid SEI is currently a highly promising technical solution. Wu et al. [29] proposed an in-situ preparation scheme for Li_2S single-ion conductor artificial SEI on the surface of metallic lithium. This interface layer has an extremely high lithium-ion conductivity, an extremely low electron conductivity, and a mechanical modulus of up to 8.66 GPa, which may help resist filament penetration under the reported conditions. Meanwhile, Li_2S has a high chemical compatibility with the sulfide electrolyte. Compared with traditional artificial interfaces such as LiF and LiI, the raw materials for its preparation are green and inexpensive, making it a more practical means for regulating the negative electrode interface (Figure 6).

Secondly, coating a thin and dense ion-conductive modification layer (such as $LiNbO_3$, Li_3PO_4, etc.) on the surface of the positive electrode particles is also a common method for interface modification. This can effectively prevent the direct contact between sulfides and oxide positive electrodes, and inhibit the oxidation side reactions at the interface. In addition, this method can buffer the volume deformation of the positive electrode, and avoiding interface failure caused by the volume change of the positive electrode. Shi et al. [30] constructed a composite electrode system by using 75% Li_2S-25% P_2S_5 (LPS) sulfide electrolyte and $LiNiMn_{0.3}Co_{0.2}O_2$(NMC) positive electrode. By constructing a 6-8 nm thick Li_2O-ZrO_2 (LZO) nano-coating layer on the surface of NMC particles, the interfacial element inter-diffusion and side reactions can be effectively inhibited, the

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interfacial charge transfer impedance can be reduced, and this provides a fundamental guarantee for the interface chemical stability of high-load positive electrodes.

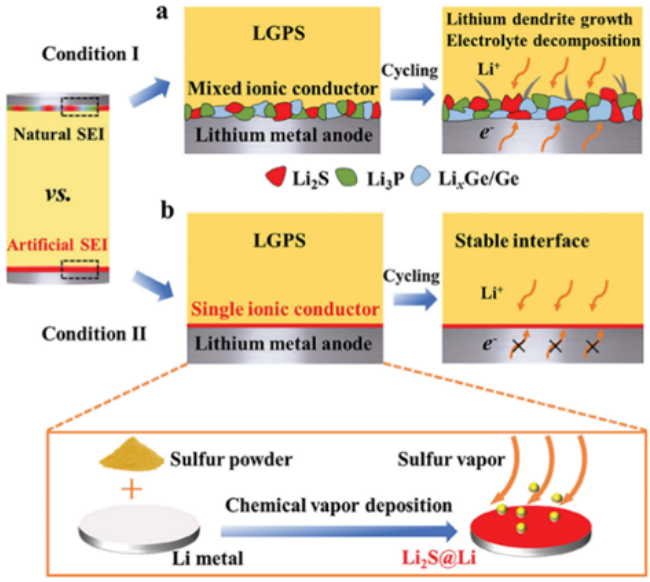


Figure 6. Contrast between spontaneously generated SEI and deliberately engineered SEI regarding Li and LGPS: a) Diagram illustrating interface mismatch between Li and LGPS; b) illustration depicting stable Li/LGPS interface following Li2S artificial SEI incorporation through chemical vapor deposition technique [29]

4. Conclusion

The failure mechanisms such as chemical/electrochemical incompatibility at the solid-solid interface, mechanical contact peeling, and lithium dendrite penetration remain the main bottlenecks restricting its large-scale application under low compression. This article provides a comprehensive overview regarding the interface failure process of sulfide all-solid-state lithium batteries alongside low-pressure stacking stabilization approaches. The primary findings are outlined below:

Interface degradation in sulfide-based all-solid-state batteries primarily involves three distinct mechanisms: Firstly, chemical-electrochemical interactions along with the space charge layer phenomenon. Secondly, volumetric shifts during cycling result in mechanical instability. Lastly, non-uniform lithium deposition triggers the formation of lithium dendrites.

To address these challenges, the following modification strategies can be adopted for optimization: (1). Material-level modification is carried out to control the volume strain of the electrode. (2). Interface engineering optimization: Build an artificial interface layer on the surface of high-voltage positive electrode particles, introduce functionalized intermediate layers (SEI), or coat an ion-conductive modification layer on the positive electrode surface.

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