

Frontiers of solid-state batteries

Jagjit Nanda, Chongmin Wang, and Ping Liu, Guest Editors

Solid-state batteries are considered the holy grail of next-generation battery technology, meeting the ever-increasing demand for energy storage that is affordable and safe, with high energy density and long cycle life. Materials and interfaces play a critical role for their eventual success and mass commercialization. This issue of *MRS Bulletin* focuses on the current state of the art of solid-state electrolytes and device architectures and provides a perspective into the various materials and interfacial challenges that limit its performance and stability.

Relevance and historical background

The appeal of solid-state battery systems is undeniable.¹ In the case of lithium-based batteries, many of the issues associated with the use of the organic liquid electrolytes can be mitigated. The removal of organic solvents can reduce flammability and the amount of combustible materials, making the system safer. A solid electrolyte layer also serves as the battery separator. This enables complete decoupling of the anode and cathode chemistries. Materials that suffer from dissolution in liquid electrolytes can be made to cycle reversibly. Most importantly, the use of Li-metal anodes becomes possible since a solid electrolyte can eliminate dendrite growth if lithium ion is the only charge carrier and the material has a high modulus.

Further, inorganic electrolytes can potentially be stable in the presence of lithium metal. In contrast, batteries with liquid electrolytes have relied on the formation of a solid electrolyte interphase to maintain stability. Liquid electrolytes are limited by their freezing and boiling points, near which the electrolytes lose their conductivities. Solid electrolytes, on the other hand, can function over a wide temperature range and their conductivities can vary continuously. This advantage becomes especially enabling at high temperatures. Solid-state battery cells can be stacked in a bipolar arrangement to form a high-voltage single cell, thus yielding a simplified system architecture.

Although the history of solid-state batteries can be traced back to the 1830s, the advantages of solid-state batteries were not fully recognized until the 1960s with the discovery of beta-alumina, a sodium-ion conductor.² This stable, highly conductive ion conductor led to the development of a commercially relevant high-temperature Na-S battery by the Ford Motor Company in the 1960s and the ZEBRA battery by the Zeolite Battery Research Africa Project group at the Council for Scientific and Industrial Research (CSIR) in Pretoria, South Africa, in the 1980s. It should be recognized that these two batteries are not literally solid-state batteries since the electrode materials were in a molten state (they are more appropriately called solid-state electrolyte batteries). In the 1970s, solid polymer electrolytes based on lithium salt-poly(ethylene oxide) complexes were discovered, which led to true all-solid-state batteries.³ The subsequent discovery in 1983 of lithium phosphorus oxynitride (LiPON), at Oak Ridge National Laboratory, resulted in the development of thin-film solid-state batteries that function at ambient temperatures with exceptional cycling stabilities.⁴

The ensuing decades saw the emergence of new solid-ion conductors with ever-increasing conductivities. Evolution of the Li-M-(P)-S-based system, which is rich in the variety of M elements that can be used, culminated in the discovery of $\text{Li}_{10}\text{GeP}_2\text{S}_{12}$ (LGPS) in 2011 with a conductivity

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of $12 \text{ mS}\cdot\text{cm}^{-1}$, which exceeds that of many organic liquid electrolytes.⁵ In the 1990s, lithium-ion conductors based on sodium (Na) Super Ionic CONductor, or NASICON-type structures offered conductivities of more than $10^{-3} \text{ S}\cdot\text{cm}^{-1}$, and the $\text{Li}_{1+x}\text{Al}_x\text{Ti}_{2-x}(\text{PO}_4)_3$ (LATP) material was commercialized.⁶ At the same time, perovskite $\text{Li}_{0.05-3x}\text{La}_{0.5+x}\text{TiO}_3$ (LLTO) was developed with a bulk conductivity of $10^{-3} \text{ S}\cdot\text{cm}^{-1}$.⁷ Weppner et al. studied garnet-like materials as solid electrolytes in the 2000s.⁸ Examples of these materials include $\text{Li}_5\text{La}_3\text{Ta}_2\text{O}_{12}$ and $\text{Li}_7\text{La}_3\text{Zr}_2\text{O}_{12}$ (LLZO), with conductivities of up to $5 \times 10^{-4} \text{ S}\cdot\text{cm}^{-1}$.

Today, interest in solid-state batteries has never been greater.⁹ Technologically, this is stimulated by the desire to pursue energy densities beyond those of lithium-ion batteries (although they continue to improve at a steady, but incremental, pace). The promise of using solid electrolytes to enable stable lithium-metal electrodes is highly coveted. Scientifically, the discovery of new solid electrolyte materials provides the needed platforms to enable technology development.

Many challenges remain to obtain solid-state batteries with performance metrics that exceed today's lithium-ion batteries. First, the electrolyte material needs to meet an array of performance requirements (**Figure 1**). Current leading solid electrolyte materials all suffer from one or more shortcomings. High conductivity sulfide electrolytes tend to have poor oxidative chemical stability, while garnet-type materials are notoriously brittle. A common challenge, however, is the difficulty of device integration. **Figure 2** shows three common configurations of solid-state batteries. A thin-film battery (Figure 2a) fabricated by vacuum deposition has been commercialized. Due to kinetic limitations, the thickness of each individual layer is limited, which restricts the maximum areal specific capacity ($\text{mAh}\cdot\text{cm}^{-2}$) of the system. In order to address this issue, three-dimensional batteries with interdigitated electrode structures have been proposed (Figure 2b).¹⁰ However, this configuration demands a conformal electrolyte layer with complex contours. Alternatively, all-solid-state batteries are

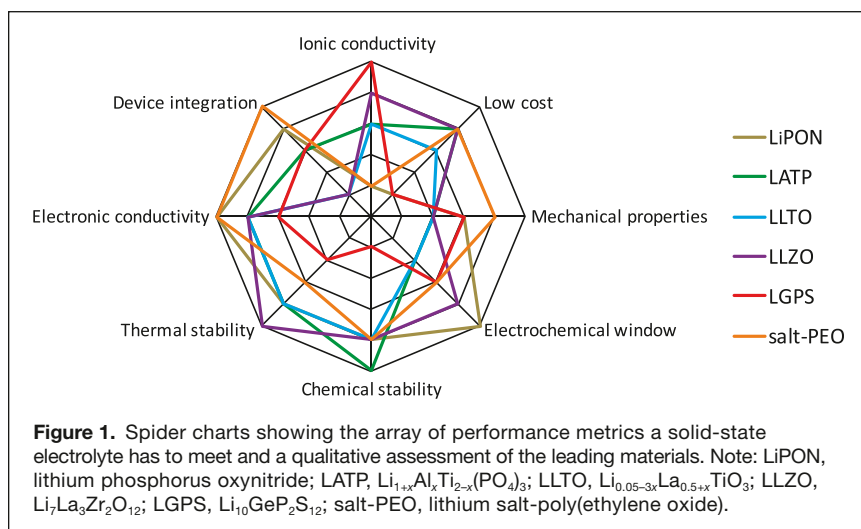
envisioned to have a structure similar to that of a lithium-ion battery. Composite electrodes (Figure 2c), composed of an active material, electrolyte, and conducting additives, are separated by a solid-electrolyte layer. This configuration dominates the research literature.¹¹ The main challenges include creating effective solid electrolyte/active material interfaces, as well as an overall reduction of the amount of solid electrolyte in the battery in order to achieve competitive energy densities.

Scientific understanding and challenges Bulk and interfacial stability

Solid-state batteries are multiscale devices, both from a material as well as a system perspective, encompassing various physiochemical and electrochemical transport phenomena occurring at multiple length and time scales.¹² For example, the secondary or aggregated transition-metal oxide cathode particles are in the size range of several microns, with primary particles being tens of nanometers or larger. Commercially available lithium foils are in the range of hundreds of microns thick, with the exception of evaporated Li films that can be much thinner. The solid electrolyte separator typically ranges in thickness between tens of microns up to the submillimeter regime.

The cell and stack size vary depending on the specific requirement and chemistry. The cell architecture has multiple material interfaces, including the Li-metal/solid electrolyte, solid electrolyte/cathode, and respective interfaces between the current collectors. Functioning solid-state batteries need to have high interfacial and electrochemical stability to enable high power and a larger voltage window.^{13,14} This requires lower area-specific resistance both at the anode (Li-metal)/solid-electrolyte interface as well as the solid electrolyte/cathode interface. Further, the interface should be electrochemically (and chemically) stable at both anodic and cathodic limit to avoid formation of unfavorable passivation or reaction layers.

A wide set of interfacial reactions between the electrode and electrolyte phases are possible, and simple thermodynamic calculations are insufficient to determine the phases that may form, and how these phases will impact interfacial resistance. The reaction layers that form may either support or be detrimental to facile ionic conduction. They may occur quickly or only after a significant number of cycles.¹⁵ They may be stable or grow over the life of the cell. Achieving and maintaining low interfacial resistance requires an understanding of the phases present on the surfaces of solid electrolytes, why these phases form, and how they evolve as a function of time, cycle number, and processing conditions. Studies suggest that high wettability between the solid electrolyte and electrode also ensures lower interface resistance.¹⁶ An ideal example is the Li metal/LLZO interface. LLZO surfaces when exposed to moist air readily form Li_2CO_3



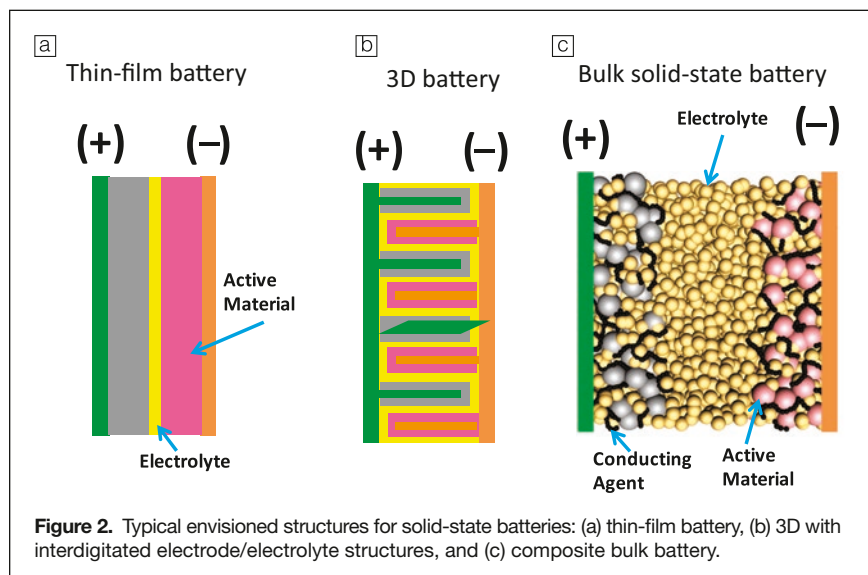
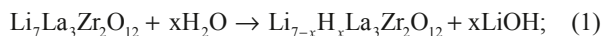


Figure 2. Typical envisioned structures for solid-state batteries: (a) thin-film battery, (b) 3D with interdigitated electrode/electrolyte structures, and (c) composite bulk battery.

via intermediates.^{15,17} One of the preferred reaction pathways shown schematically in **Figure 3** proceeds via a two-step process that involves protonation of LLZO (Li^+/H^+ exchange) and formation of LiOH as an intermediate product.¹⁵ The two-step reaction can be described as:



The contamination layer on LLZO is composed primarily of Li_2CO_3 , LiOH, and adventitious carbon that contributes to the high interface resistance. Within experimental accuracy, depth profile x-ray photoelectron spectroscopy (XPS) studies on surface-contaminated LLZO show carbonate rich layers up to 40 nm followed by a buried LiOH layer. Recent studies have reported a surface-coating-free approach toward reducing the interfacial resistance by wet polishing followed by

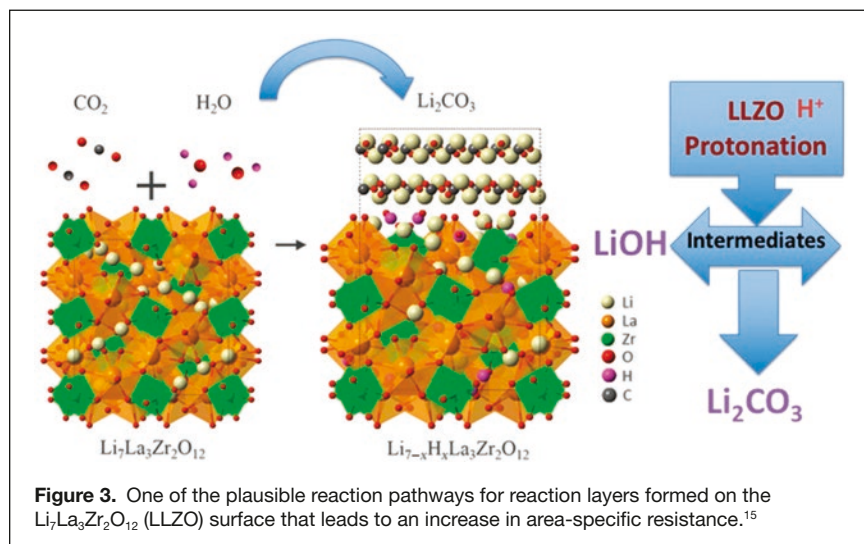


Figure 3. One of the plausible reaction pathways for reaction layers formed on the $\text{Li}_7\text{La}_3\text{Zr}_2\text{O}_{12}$ (LLZO) surface that leads to an increase in area-specific resistance.¹⁵

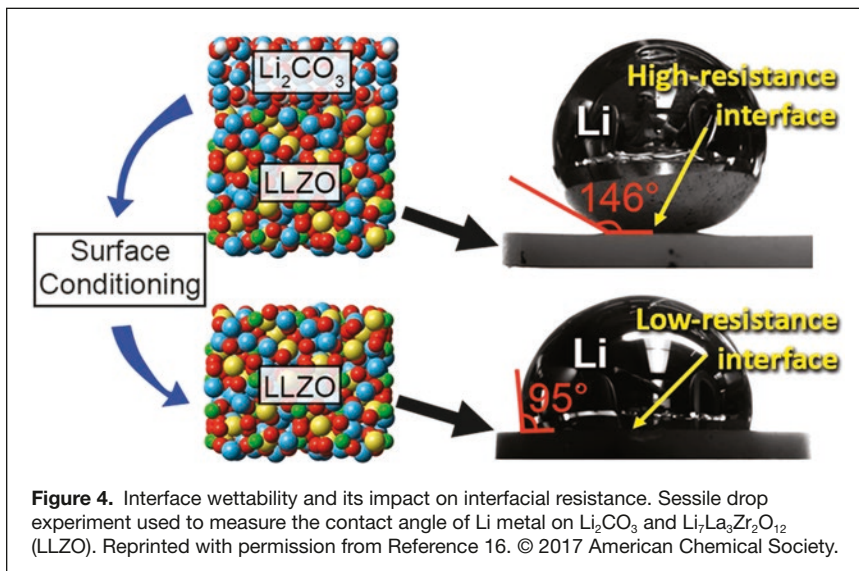
heat treatment under an inert atmosphere.¹⁶ An interfacial resistance as low as $2 \text{ ohm}\cdot\text{cm}$ was reported using this process. The removal of the contamination layer on LLZO also improved wetting of Li metal that led to a decrease in the interfacial resistance as demonstrated by contact angle measurements using molten Li-metal under various LLZO surface conditions (**Figure 4**).¹⁶ This example demonstrates the close interplay between surface composition (conditioning) and wettability toward achieving lower interface resistance.

In situ studies and characterization

Much progress has been made in developing *in situ* tools based on electrons, ions, and photons for directly observing the structural and chemical evolution of battery materials under dynamic operating conditions. Unlike *ex situ* studies that involve unexpected reactions due to the removal of the samples from their native and reactive environment, *in situ* electrochemical characterization mimics the true environment that a real battery experiences.^{18,19} Essentially, solid-state battery configurations using solid electrolytes provide a convenient way for probing the structural and chemical evolution of electrode materials.

For the all-solid-state configuration, a device used for *in situ* studies may be related to the solid-state system as a battery itself or as a platform for *in situ* transmission electron microscopy (TEM) studies of a battery system. Brazier et al.²⁰ developed the first cross-section preparation method for an all-solid-state Li-ion “nanobattery” for *in situ* TEM observations. The fundamental concept of this configuration is to use a focused ion beam (FIB) to make a “nanobattery” from an all-solid-state battery prepared by pulsed laser deposition (PLD). Using a similar configuration, Yamamoto et al.²¹ observed *in situ* changes in electric potential in an all-solid-state Li-ion battery using electron holography. They mapped the two-dimensional potential distribution resulting from movement of lithium ions near the positive-electrode/electrolyte interface. Meng et al.²² probed dynamic phenomena in an all-solid-state nanobattery based on imaging and electron energy-loss spectroscopy (EELS). Also Fawey et al.²³ used an optimized FIB-based approach to prepare a micron-sized battery for *in situ* TEM studies in addition to starting from a powder-based all-solid-state fluoride-ion battery.

Imaging of a fabricated nanobattery inside a TEM allows for real-time atomic-scale observations of the dynamic evolution of the interface and internal structure of the electrode during operation of the battery with unprecedented spatial resolution. This allows for an atomic and nanoscale understanding of the mechanisms



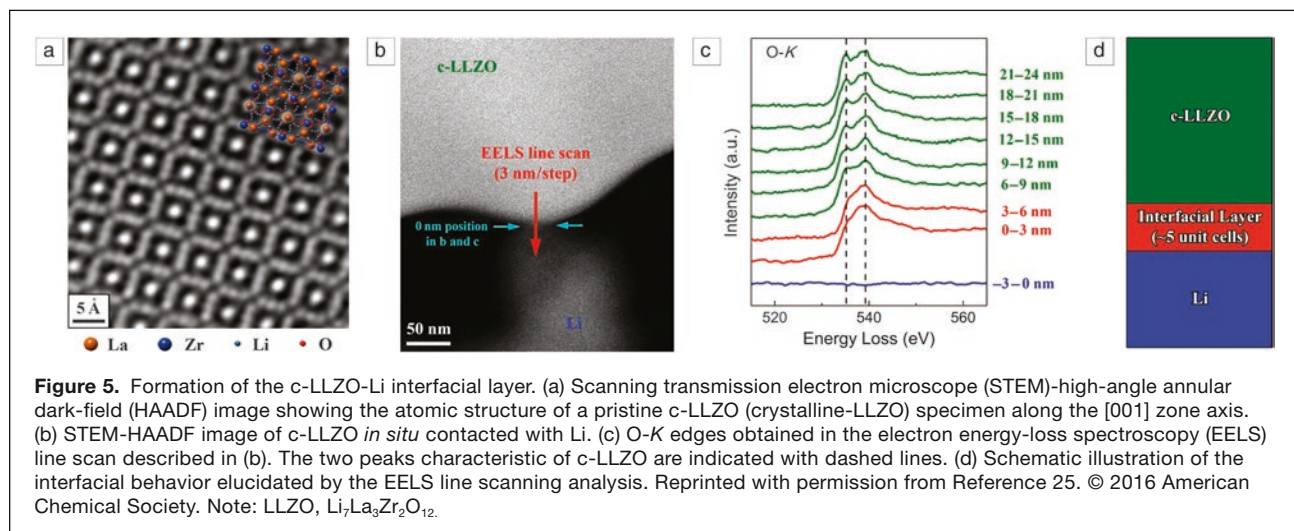
associated with the following: (1) Li-ion insertion and extraction mechanisms during the electrochemical cell operation; (2) the nature of the solid electrolyte interphase (SEI) layer between the electrode and the electrolyte, along with the movement of the SEI layer inside the electrode during charging/discharging; and (3) the change in the composition/structure of the SEI layer, along with the orientation/morphology of the nanostructured electrodes. Overall, the all-solid-state nanobattery provides a platform for coupled imaging, diffraction, and spectroscopy for comprehensive structural and chemical analysis of nanobatteries under typical battery operating conditions and offers the possibility of high spatial resolution imaging.

The behavior of functional interfaces is a crucial factor in the performance and safety of energy-storage and conversion devices. Indeed, solid electrode–solid electrolyte interfacial impedance is now considered the main limiting factor in all-solid-state batteries. Wang et al.²⁴ presented a new approach to

conducting *in situ* scanning transmission electron microscopy (STEM) coupled with EELS in order to uncover the unique interfacial phenomena related to Li-ion transport and its corresponding charge transfer. Their approach allowed quantitative spectroscopic characterization of a galvanostatically biased electrochemical system under *in situ* conditions. Using a $\text{LiCoO}_2/\text{LiPON}/\text{Si}$ thin-film battery, an unexpected structurally disordered interfacial layer between the LiCoO_2 cathode and LiPON electrolyte was discovered to be inherent to this interface without cycling. During *in situ* charging, spectroscopic characterization revealed that this interfacial layer evolved to form highly oxidized Co ion species along with lithium-oxide and lithium-peroxide species. These findings suggest that the mechanism of interfacial impedance at the $\text{LiCoO}_2/\text{LiPON}$ interface is caused by chemical changes rather than space-charge effects. Insights gained from this technique will shed light on important challenges of interfaces in all-solid-state energy-storage and conversion systems and facilitate improved engineering of devices operated far from equilibrium.

Ma et al.²⁵ used *in situ* STEM and EELS (Figure 5) to reveal the formation of a Li-carbon–LLZO interface. They found that upon contact with Li metal, the LLZO surface is reduced, accompanied by the simultaneous implantation of Li^+ and resulting in a tetragonal-like LLZO interphase that stabilizes at an extremely small thickness of around five unit cells. This interphase effectively prevented further interfacial reactions without compromising the ionic conductivity.

One of the major concerns for *in situ* TEM studies of batteries is the electron-beam-induced artifact, typically beam-induced amorphization. To mitigate this problem, the electron-beam condition, especially the electron dose rate,



needs to be carefully calibrated and controlled such that no obvious beam damage occurs. Cryo-electron microscopy techniques, mostly developed for biological science, can also be used to mitigate the beam effect, which has been demonstrated for the case of probing into the SEI layer.^{26,27} Ideally it would be expected that an integrated device will be developed that will allow control of both temperature and cycling of the battery, equivalent to a same platform combination of *in situ* and cryo-microscopy.

In this issue

This issue focuses on recent developments in solid ion conductors and the various surface and interfacial challenges that need to be addressed for enabling solid-state batteries. In their article in the issue, Ceder et al.²⁸ discuss first-principles modeling of ionic conductivity and interfacial reactivity to understand the intrinsic performance and limitations of solid electrolytes. Using *ab initio* molecular dynamics, ionic conductivity can be predicted with reasonable accuracy. Thermodynamic models for electrochemical stability and interfacial reactivity have been used to establish the intrinsic voltage limits at which solid electrolytes can operate in solid-state batteries.

The Dudney²⁹ article highlights the current status of efforts to stabilize the Li-metal anode, including strategies for mitigation of Li-metal dendrite growth and processing methods. Hallinan et al.³⁰ report on the current status of polymer electrolytes, including compositions and the mechanistic understanding of ion transport mechanisms in composite electrolytes. Wynn et al.³¹ provide a comprehensive review of *in situ* and *in operando* methodologies that are currently under development for solid-state interfaces and offer the potential to describe the dynamic interfacial processes that serve as performance bottlenecks for solid-state batteries. Finally, Hao et al.³² provide their perspective on device architecture of solid-state batteries and discuss various fabrication approaches for achieving intimate interparticle and interlayer contact that are critical for solid-state battery performance.

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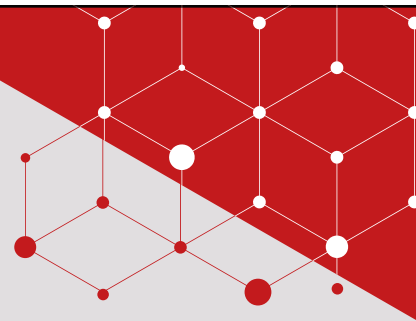
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