A Proposed New Paradigm for Understanding and Preventing Li Metal Penetration through Solid Electrolytes

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Context & Scale

A promising future technology for higher energy-density batteries is use of Li or Na anodes with ceramic solid electrolytes (SEs). These batteries have not been practical to date, in large part because Li/Na dendrites readily penetrate through SEs, leading to short circuits, even though the Li and Na shear moduli are at least 1-2 orders of magnitude below those of the SEs. This Perspective proposes a solution to dendrite penetration that is completely new to the battery field, one that we believe will unlock new research areas for improved SEs. Our approach comes from the observation that water readily penetrates through steel and other hard solids in a process called stress corrosion cracking (SCC). The driving force for SCC is generated at the crack tip by electrochemical reactions in the presence of high tensile stress. We propose that Li penetration through SEs is analogous: via crack growth driven by Li reactions in SE regions of high tensile stress. The SCC problem was solved decades ago for metals, glasses, and ceramics by putting their surfaces into states of residual compressive stress sufficiently high to inhibit crack propagation. A

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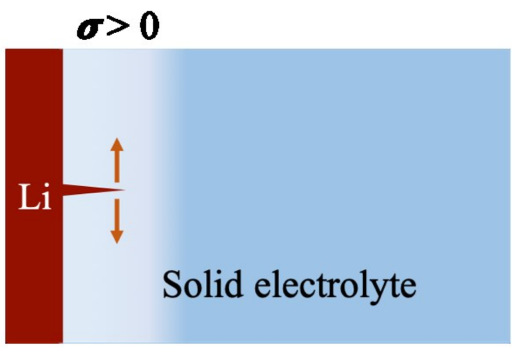
similar approach could be viable for SEs if the required residual compressive stress does not strongly hinder Li ion diffusion. In this work we perform molecular dynamics calculations that show only a modest impact of even very high (10 GPa) compressive stresses on Li ion diffusion in a typical SE, potentially enabling our approach.

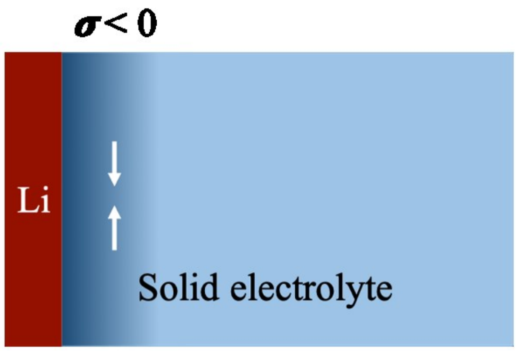
Abstract

The use of lithium (Li) or sodium (Na) metal anodes together with highly ion-conductive solid electrolytes (SEs) could provide batteries with a step improvement in volumetric and gravimetric energy dendrites. Unfortunately, these SEs face significant technical challenges, in large part because Li and Na dendrites can penetrate the SEs, leading to short circuits. The ability of a material with a low shear modulus (Li or Na metal) to penetrate through a ceramic is surprising from the point of view of models widely used in the Li battery field, which indicate that a SE with a shear modulus more than double that of Li or Na should prevent dendrite penetration. We introduce a concept new to the battery field for suppressing formation and penetration of lithium dendrites through SEs by putting the SE surfaces into a state of residual compressive stress. For a sufficiently high compressive stress, cracks have difficulty forming, and cracks that do form are forced to close, inhibiting dendrite penetration. This approach is widely used to solve commercially important stress corrosion cracking problems in metals and static fatigue problems in ceramics and glasses (e.g., Gorilla Glass). However, the technique will not be useful for SEs if the Li ion transport rate through a SE is substantially reduced when it is in compression. Our molecular dynamics calculations for Li ion transport through a common SE demonstrate that the introduction of even very high residual compressive stresses (~10 GPa) has only a modest effect on Li ion transport kinetics, suggesting that the approach is viable and capable of providing a new paradigm for developing high-performance and stable SEs.

TOC Graphics



Tensile surface stress promotes Li dendrite growth



Compressive surface stress prevents Li dendrite penetration

Introduction

The use of Li or Na metal negative electrodes (anodes) could provide a substantial increase in the gravimetric and volumetric energy density for Li batteries. However, their use in high capacity rechargeable batteries has been precluded[1], in large part because of the growth of Li filaments (loosely called "dendrites"). These filaments provide a surface for degradation reactions, and they may become electrically isolated. For the case of dendrite penetration into solid electrolytes, which occurs readily[2], they can induce mechanical failure and fracture of solid electrolytes, and if they reach the positive electrode, they will cause a short circuit, potentially leading to a fire [3-7].

The battery community has taken several approaches to addressing the dendrite penetration problem[8,9]. Monroe and Newman and followers [10-12] proposed an electrochemical-mechanical model in which a Li protrusion (proto-dendrite) is pressed conformally, or nearly conformally, against a solid polymer separator. These models compare reaction current density at the tip of the protrusion (dendrite penetration) with current density at its base and find that a separator with a shear modulus at least double that of Li metal could prevent dendrite penetration. However, neither suppression of dendrite penetration in liquid electrolyte cells under moderate stack pressures[13,14], nor penetration through solid electrolytes with very high shear moduli[2,15]—cases that do not involve solid polymer separators—can be readily explained with this paradigm. We attribute this discrepancy to a combination of the model not incorporating Li creep; ignoring internal defects in the solid electrolyte; and assuming near-conformal contact, which is not realized for interfaces between Li metal and either thin commercial separators[13] or solid electrolytes[15]. Because only a small fraction of the Li metal and solid electrolyte surfaces are in physical contact, GPa-level hydrostatic pressures[16] in Li cannot build up against solid electrolytes because Li relaxes, by deformation and creep, into non-contact regions that may be at a considerable distance from the contact point. Thus, we believe that contact mechanics between Li and a solid electrolyte cannot be properly modeled on a scale of a single penetrating dendrite. In addition, internal defects, such as pores, cracks, and grain boundaries in solid electrolytes can trap electrons and nucleate metallic Li, even ahead of the Li-metal front[17].

Chemical and physical modifications to the Li-separator interface, such as introduction of nanostructures or coatings or by modification of the electrolyte[18-32], have seen some success in inhibiting dendrite growth, while other approaches have focused on developing new solid electrolytes[33] and special Li hosts[34,35] or on controlling the temperature[36]. Nevertheless, to date there are no commercially available high capacity rechargeable Li metal batteries that operate under a current density that is comparable to that in liquid electrolytes, in significant part because of the dendrite penetration problem. This Perspective proposes a new paradigm for mitigating dendrite-induced short circuits.

New Approach to Suppress Lithium-Dendrites Induced Cracking

Our proposed approach is based on an analogy to stress corrosion cracking (SCC)[37-40], which has been studied and largely solved in the corrosion field. SCC occurs when the surface of a component is chemically (usually electrochemically) attacked at a defect or heterogeneity[41]. The result is the formation or extension of an incipient crack that can grow if and only if the crack tip is in a sufficiently high state of tension. SCC crack growth occurs under the combined effect of a chemical reaction plus surface tensile stresses that are above some threshold value but below the level required for fast crack propagation[42,43]. SCC has led to catastrophic results, such as the collapse of the Silver Bridge[44,45] across the Ohio River. In that case, local rusting caused stress corrosion cracks that followed grain boundaries in the steel. Although SCC is often thought of as occurring primarily in metals, it can also occur in glasses, ceramics, and quartz, where the process is known as static fatigue [46-51].

Since the attacking medium in SCC is usually water, relative shear strength[10,11] clearly plays no role, so the fact that soft Li metal penetrates through extremely stiff ceramic solid electrolytes is not surprising if SCC is the mechanism. Furthermore, hydrostatic pressures in the Li are not required, since the SCC driving force comes from chemical reactions in regions of tensile stress in the solid electrolyte. The proposed analogy between water penetration and Li metal penetration can be visualized in **Figure 1**. **Figure 1a** shows stainless steel that has been

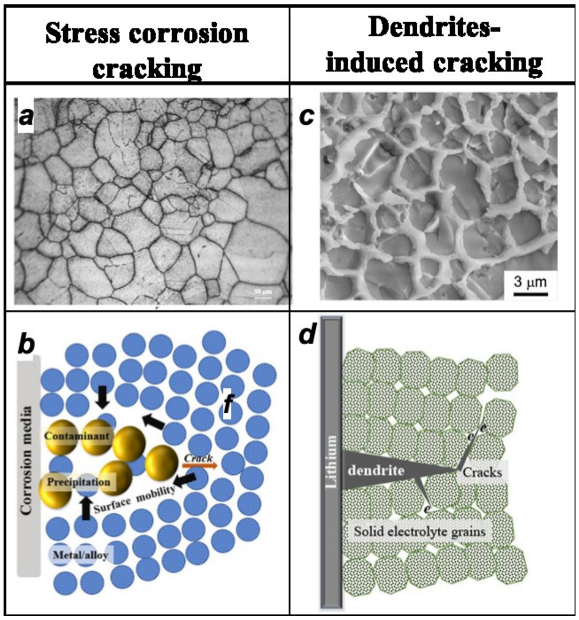


Figure 1: SEM images of (a) intergranular cracking in stainless steel (b) the intergranular penetration of LLZO by Li. Schematics of cracking propagation caused by SCC (c) and lithium plating in LLZO (d). the corresponding SCC mechanism.

attacked by water via SCC[52], where intergranular cracks were observed during aging at 550 ˚C. The formation of chromium carbides along the grain boundaries, which is facile at elevated temperature, results in creation of a chromium-depleted region, exacerbating the susceptibility to intergranular corrosion when tensile stresses are present. **Figure 1c** shows images of intergranular penetration of LLZO[2] by Li metal, forming dendrites as the crack propagates.[2] As illustrated in **Figures 1b** and **1d**, both processes involve corrosion/electrochemical environments together with the presence of heterogeneities[41], including grain boundaries, contaminants or precipitates. We suggest that, in analogy with SCC, local residue tensile stresses promote Li dendrite propagation via crack propagation.

The approach that often solves SCC problems is putting the surface into a state of residual compressive stress. For example, the crack resistance of Gorilla Glass is due to exchanging smaller sodium atoms near the surface with larger potassium atoms[53].

We are not aware of any spatially resolved surface stress measurements in solid electrolytes. However, large residual stresses, both tensile and compressive, can be expected from formation processing[54], and experiment shows that when tensile, these stresses can result in highly brittle products[55]. Local tensile residual stresses can also be generated from heterogeneities as well as from machining or polishing[56-59]. Thus, solid electrolytes may be readily susceptible to SCC (or static fatigue) as well as brittle fracture.

However, the analogy between solving SCC and solving Li dendrite penetration is imperfect because a solid electrolyte must also maintain sufficient ionic conductivity after the compression is introduced, a factor that is irrelevant for something like Gorilla Glass. Previous work has shown that internal stresses can have a significant impact on ionic conductivity[60-66], and it is possible that putting the solid electrolyte into compression could substantially hinder ion mobility.

Evaluation of the Impact of Stress on Li Diffusion

To evaluate the effect of a hydrostatic compressive stress on Li diffusion kinetics, molecular dynamics (MD) simulations were performed as described in the Methods section. Results are shown in **Figure 2, left**, which were calculated at 1100K to accelerate the diffusion process. The Li self-diffusivity, D, was fitted to the linear region of the root mean square distance (RMSD) with simulation time. The diffusivity was then plotted as a function of the average hydrostatic pressure. The simulation results at 1100K (orange dots, **Figure 2, right**) show a peak diffusivity at 2GPa tensile stress. In order to compare with experiments at room temperature, the simulated diffusivity was extrapolated to 300K based on the experimentally measured activation energy of 0.38 eV[67], a value that is insensitive to strain change according to density functional theory (DFT) calculations[68]. It can be seen from the blue dots, **Figure 2, right,** that the simulated/extrapolated room temperature Li diffusivity at zero stress is ~0.5×10^{-12} m^2/s, which is within the range of

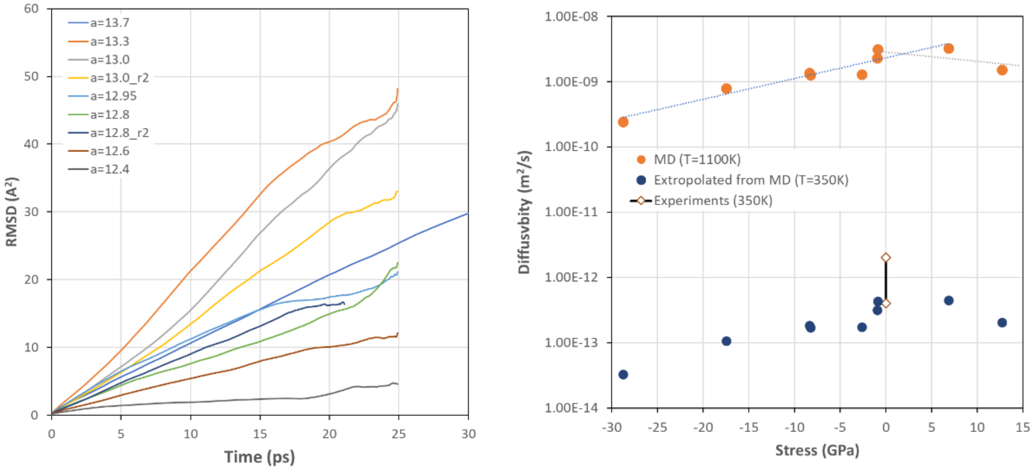


Figure 2: The simulated (left) average Li RMSD vs. simulation time with varying lattice parameters, (right) the Li self-diffusivity as a function of stress (positive means tension).

experimental values $0.4-2\times10^{-12}$ m^2/s[67], supporting the MD simulation results. It is not surprising to see that the Li diffusivity peaks around 2GPa tensile stress, because fast ionic conductivity in LLZO is enabled by low-barrier concerted ion migrations, which are triggered by the strong ion-ion Coulomb interactions at unique ion configurations. While compression shrinks the free volume available for Li ion mobility, high tension may also reduce the Coulomb interactions required for fast ion conductivity.

Although the Li diffusivity decreases under compression, even at 10GPa compression the diffusivity is still 40% of its value at zero stress. This difference is even less than the variation of the experimental data at zero stress. Consistent with our results, we note that the experimentally measured Li ion conductivity in LLZO showed minimal changes under compressive residual stresses between 12.5Mpa and 50Mpa[69]. Therefore, we conclude that even very large compressive stresses will not have a large impact on Li ion diffusion over short distances.

Methods for Applying Compressive Stress

As mentioned above, SCC can only occur when the surface is in tension, so a highly successful and widely used strategy for preventing SCC has been to put the component's surface into compression. If this residual compressive stress is high enough, cracks are difficult to form and grow, and cracks that exist are forced to close. Among the most widely used techniques to introduce a surface residual compressive stress in metals is shot peening[70-73], which is a cold working process where large numbers of small hard particles (metallic, ceramic or glass) impinge on the component with sufficient force to deform it plastically and put the surface into compression[74,75]. For brittle materials such as ceramics and glasses, other techniques have been developed for commercial application. Among them are laser shock peening (LSP), which can give compressive stresses of up to 500 MPa in aluminum oxide[76]; ion implantation[77,78]; and ion exchange[47,79] (which is used to make Gorilla Glass[53]). The latter techniques can introduce compressive residual stresses as high as 10 GPa in aluminum oxide [78]. We next consider each of these approaches, except for shot peening, for use with LLZO.

LSP introduces laser plasma-driven shock-waves into a material, typically using a plasma-confining medium (e.g., glass or water) to increase the peak pressure of the shock wave. A sacrificial layer is often used to protect the treated sample from laser ablation. Shukla et al[76] demonstrated that his LSP-treated a-Al_2O_3 had a biaxial compressive residual stress of several hundred MPa that extended to a depth of up to 1.2 mm from the surface.

Ion exchange[80] has also been used to introduce compressive stresses at the surface by exchanging alkali (or sometimes alkaline-earth) ions in the original glass/glass-ceramic with larger ones from a molten salt bath. The introduction of large ions near the surface induces a volume increase, generating a compressive stress in the ion-exchange region. The final mechanical properties are dependent on the stress level at the surface and the depth of penetration of the larger ions.

Unfortunately, the uses of these strengthening strategies for preventing Li dendrite penetration through solid electrolytes face potentially severe constraints. In the case of ion exchange, only a limited selection of ions can be introduced near the SE surface in a wet process. Also, ion exchange strongly favors the exchange of monovalent ions—replacing the Li in this case—which would change the chemical identity of the solid electrolyte and, likely, inhibit Li ion mobility in the exchanged region. While LSP avoids these problems, it generally creates heterogeneous surface damage that could increase the interface resistance; it can also create local tensile stresses[76].

Furthermore, the range of stresses introduced by LSP, up to hundreds of MPa, is much lower than what is possible with other techniques.

The better alternative, in our estimation, is ion implantation, which can be used to introduce residual surface compressive stresses with control of stress level, depth profile, crystallinity, and chemistry in the near-surface region (10 to 1,000 nm). It has been used commercially to strengthen polymers, glasses and ceramics for decades to solve stress corrosion cracking (SCC) or static fatigue, but it has not been applied to SEs, to our knowledge. An advantage of ion implantation is the large number of chemical, structural, and physical states that can be created, including metastable and non-equilibrium states, for nano/mesoscale tailoring the surface structure of SEs.

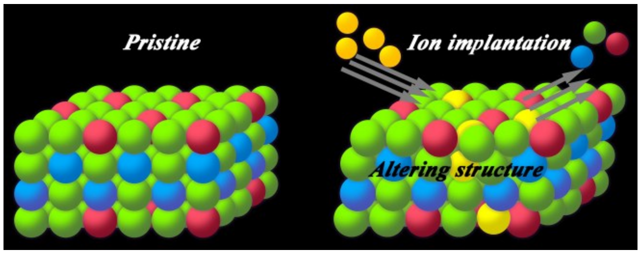


Figure 3: Scheme of using ion implantation to alter structure of solid electrolytes.

Energetic implanted ions can include transition metal ions, halide ions, rare gas ions, and even lithium ions, which can modify the surface structure—including creating local disorder—leading new mechanical properties for solid electrolytes, as illustrated schematically in **Figure 3**. With its control of dopant species, concentration, and spatial distribution (implantation depth), ion implantation has also been widely used in modifying electronic properties of semiconductors for the microelectronics industry, as well as strengthening of metals and non-conducting ceramics[81]. Recent work has shown a positive impact of using ion beam modification on the ionic conductivity of polymer electrolyte films due to increased charge carries and dielectric constant.

Finally, we note that introduction of compressive residual surface stresses would be expected to make any SE less brittle and, thus, easier to handle in a commercial environment. We expect that implantation of solid electrolytes will create materials with new properties that can be tailored to improve those of solid-state ion-conducting materials.

Conclusion and Perspective

In summary, we have proposed a new paradigm for inhibiting dendrite penetration through solid electrolytes: putting the SE surfaces into a state of residual compressive stress, which can suppress the crack formation that accompanies dendrite penetration. We have performed MD simulations to demonstrate that the introduction of even very high ($\sim$10 GPa) hydrostatic compressive stresses into LLZO has only a modest impact on Li diffusion kinetics, enabling our proposal. Our approach is based on its success in solving stress corrosion cracking and static fatigue problems in metals, ceramics, and glasses in commercial products. In addition to inhibiting dendrite penetration in solid electrolytes, compressive stresses on the order of GPa will improve their fracture toughness and will tend to suppress lithium plating inside of solid electrolytes because of an increased overpotential. While there are a number of techniques that can be used commercially to introduce compressive residual stresses, we suggest that ion implantation will be the most useful for this purpose, as it may provide a new avenue for developing high-performance and stable solid electrolytes.

METHODS

A periodic cubic cell of $Li_{24}La_{24}Zr_{16}O_{96}$ was simulated using GULP software and implemented in Materials Studio with a force-field including the long-range Coulombic potential, the short-range Buckingham potential, and a core–shell polarizable potential for O atoms following the parameters provided in[82]. The simulation cell length was varied from 12.4 Å to 13.7 Å, and the root mean square displacement (RMSD) of Li^+ ions was tracked during NVT (fixed cell) dynamics.

AUTHOR CONTRIBUTIONS

S.J.H. and C. B. proposed the idea and concepts. Y.Q. did the calculations. All authors contributed equally to the writing of the manuscript.

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