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# **Engineering Electrodeposition for Nextgeneration Batteries**

#### by Stephen T. Fuller, Yonglin Huang, Ruixin Wu, Fudong Han, J. X. Kent Zheng, and Natasa Vasiljevic

#### Electro-Plating/Stripping: The "Holy Grail" Anode Chemistries for Batteries

The redox reaction  $M^{n+}_{(sol.)} + ne^- \leftrightarrow M_{(s)}$  has been utilized since ancient times.<sup>1</sup> This utilization is due, in part, to the simplicity of the reaction and the ubiquity of the materials involved. Recently, there has been a resurgence of interest from the battery research community in the development of metal anodes that rely solely on this type of simple metal redox reaction, also known as the *plating/ stripping* or, interchangeably, the *deposition/dissolution* reaction in the electrochemistry context. The metal anodes feature simple redox chemistry by design and remarkably improved energy density (Table I). Metal electrodes of this kind, especially Li, have been dubbed the "Holy Grail" for battery anodes.

Li metal was first discovered as a possible anode for batteries over a century ago by Lewis and Keyes.<sup>2</sup> Once an organic electrolyte was developed that prevented the direct reaction of lithium with the electrolyte, primary Li metal batteries began to be commercialized in the late 1960s using various cathodes.<sup>3</sup> Initially, the transition to secondary Li batteries was focused on finding a cathode material that could reversibly intercalate and de-intercalate Li<sup>+</sup>. Li metal batteries with TiS<sub>2</sub> and MoS<sub>2</sub> cathodes were deployed commercially in the late 1970s and 80s but were quickly recalled due to the discovery of fire incidents due to metal shorting from dendrite formation on the anode.<sup>4</sup>

The academic community then turned to the graphite anode to develop the rocking chair battery. A rocking chair battery uses a host material that allows reversible intercalation and de-intercalation of the metal cations in the anode (Fig. 1A). This process is in stark contrast to a plating/stripping metal anode where a new solid phase is created through nucleation and growth processes (Fig. 1B and Fig. 2). Driven by multiple possible kinetic heterogeneities,<sup>5</sup> such processes oftentimes result in non-compact deposits that are prone to aggressive outward propagation in battery cells.

Conversely, examples of electrodeposition of metals were first reported at the turn of the 19th century, and significant developments were made in the field in the latter half of the 20<sup>th</sup> century when industrial electrodeposition became important.<sup>6</sup> The morphological evolution of metal electrodeposits turned out to be a critical issue for using metal anodes in batteries. The non-compact growth modes create a variety of fading mechanisms and safety risks (Fig. 2B). As far as we know, some of the first reports on using electrodeposition principles in batteries were in the early 1960s, which focused on limiting dendritic growth for the purpose of secondary zinc alkaline batteries.<sup>7</sup> Since then, there have been other works focused on tuning the electrodeposition morphology to improve battery cyclability.<sup>5</sup> A demand for higher energy density batteries than conventional intercalation-type rechargeable batteries has sparked further interest in electrodeposition (plating/stripping anodes) over the last decade.

In addition to the interest in raising energy density by utilizing a lithium metal anode, there has been renewed interest in the expanding library of batteries where the anode can be operated based on a metal plating/stripping mechanism to reduce the manufacturing cost of the anode. For example, in addition to the more reactive alkaline and alkali-earth metals, the plating/stripping reaction of metals such as Al, Mn, Fe, Zn, Cu, and Sn have all attracted research interest aimed at integrating them into next-generation batteries as anode materials (Table I).<sup>8</sup>

#### The Challenge of Attaining "Close-to-Unity" Reversibility

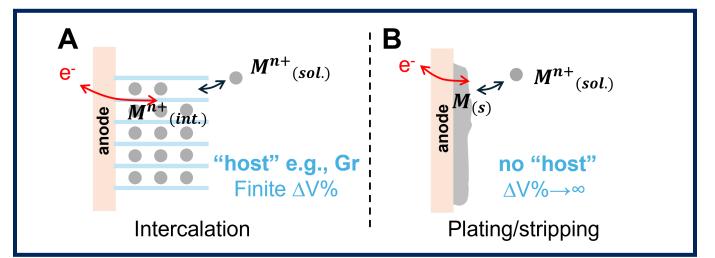
Coulombic efficiency is a key metric to determine the feasibility of these batteries, and it has been calculated that this metric must be above 99.9% for metal anode batteries to have serious commercial viability.<sup>9</sup> The deviation of this metric from 100% is known to result from two possibilities: (1) chemical degradation, which involves the reaction of the metal anode to form a solid electrolyte interphase (SEI); or (2) metal orphaning, which involves physical disconnection of fragments of the metal anode from the current collector. The academic community is beginning to accept that the latter possibility plays the most important role in coulombic efficiencies below 100%.<sup>10</sup> The past decade of research has focused primarily on limiting the dendritic growth of the batteries during the charging process through

(continued on next page)

Table I. Key Theoretical Figures-of-Merit for Battery Anode Chemistries*						
ANODE CHEMISTRY	LOG ABUNDANCE (ppm)	CAPACITY (Ah/mL)	CAPACITY (Ah/g)	CELL VOLTAGE EST. (V)	ENERGY DENSITY (Wh/mL)	ENERGY DENSITY (Wh/g)
Graphite	N/A	0.8	0.3	4	3.2	1.2
Li	1	2.0	3.9	4	8.0	16
Na	4	1.2	1.2	3	3.6	3.6
AI	5	8.0	3.0	2	16	6.0
Mn	3	7.4	1.0	2	15	2.0
Fe	5	7.9	1.0	1	7.9	1.0
Zn	2	5.8	0.8	1.5	8.7	1.2

\*Metal anodes outperform the current intercalation-based graphite anode in almost every aspect, except for the extraordinary cycling stability of the graphite intercalation chemistry. Other metals emerge as promising energy storage materials complementing the dominant Li-based technology that is limited by Li's relatively low earth-crust abundance. Li sets itself apart by its gravimetric energy density a few times to one order of magnitude higher than other metal anodes. This explains Li's key role in high-end portable energy storage, where mass is the main design consideration. By contrast, other metals, in general, exhibit comparable or even higher volumetric energy density than Li. This means these alternative chemistries may find applications in stationary energy storage or portable storage where volume is more important than mass.

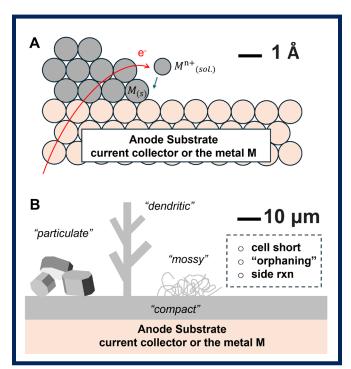
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**FIG. 1.** Electrochemical mechanisms of rechargeable battery electrodes. (A) An intercalation electrode: charged ions are reversibly inserted into / de-inserted from a host material (e.g., graphite, whose layered structure facilitates ion storage and transport) during discharge-charge cycles. This reversible reaction (e.g.,  $Li + 6C \leftrightarrow LiC_{\theta}$ ) is associated with a finite volume change  $\Delta V\%$ . (B) A metal electrode utilizing the plating/stripping reaction. In the absence of the "host," the charged ions (i.e., normally metal cations dissolved in a liquid solution) are reduced and converted to a solid phase upon charging. The dissolution of the metallic solid phase happens upon discharging. The plating/stripping reaction (e.g.,  $Li^+ + e \leftrightarrow Li$ ) is, in theory, associated with an infinite percentage volume change  $\Delta V\%$ . While a significant amount of mass/volume can be saved by removing the host, the nature of the plating/stripping reaction creates the challenge of precisely controlling such dynamic plating/stripping processes inside battery electrodes.

a variety of processes including electrolyte engineering, artificial SEI, and even modifications to the structure of the bulk metal foil.

The fundamental aspects involved in understanding and controlling the electro-plating/stripping morphology have been extensively discussed in a few prior reviews.<sup>5,11-13</sup> Here, we highlight two future



**FIG. 2.** Nucleation and growth of electrodeposits. (A) Electrons flow from the electrode surface into the orbitals of metal cations. The chargeneutral metal atoms nucleate—either epitaxially (shown in the schematic diagram) or not—on the electrode surface. (B) As the deposition proceeds, the nuclei develop into various possible morphologies or a mixture of them. It is desirable that the morphology stays compact and uniform during the plating/stripping. A porous morphology results in multiple issues causing irreversibility in plating/stripping (e.g., metal "orphaning," side reactions, etc.) and even fatal battery shorts.

research avenues that could be vital to advancing the viability of metal anode batteries in addition to the ongoing research on limiting dendritic growth during battery charging.

Use of a three-dimensional, nonplanar current collector has been shown to increase the coulombic efficiency of the metal battery to >99% at high areal capacities ~10 mAh/cm<sup>2</sup>. Rational design (e.g., focusing on the electrical conductivity and interfacial chemistry of the nonplanar architecture) is essential to achieving high reversibility.<sup>14</sup> Although these current collectors decrease the potential energy density, they could serve as a short-term solution for Li metal anode batteries. More research is needed on finding nonplanar current collector networks that are optimized for the length scale of the metal electrodeposits to maximize coulombic efficiency.

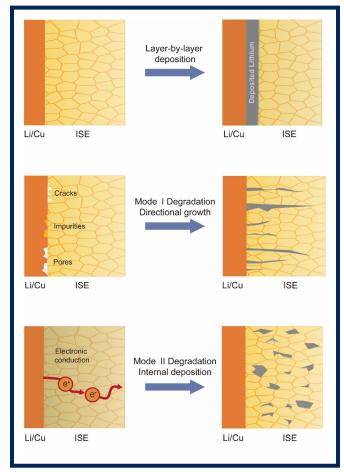
The other aspect that will be important for batteries is understanding electrodeposition and stripping in new solvent systems, such as waterin-salt electrolyte systems, ionic liquids, and deep eutectic solvents.<sup>15</sup> These electrolytes are nonflammable, have high ionic conductivity, and have higher electrochemical stability windows than conventional aqueous or organic electrolytes. Understanding how the properties of the deposits change as a function of the solvent system will have large implications for metal anodes outside of Li.

#### **Electrodeposition in the Solid State**

Lithium metal anodes (LMAs) are considered the ultimate solution for high-energy batteries.<sup>16,17</sup> While LMAs have already been successfully applied to thin-film solid-state batteries,<sup>18</sup> the utilization of LMAs in high-areal-capacity, bulk-type solid-state cells faces several challenges such as interfacial instability, dendritic growth, and morphological instability.19,20 As Li electrodeposition is the very first process on the anode during the initial charge of solid-state Li metal batteries and occurs every subsequent charge/discharge cycle, it plays a critical role in governing the performance and degradation of batteries, especially for anode-free batteries where there is no Li on the anode prior to Li plating. In this section, we will briefly overview the physicochemical concepts that describe the electrodeposition of Li in inorganic solid electrolytes with a focus on the growth behavior. While the discussions are primarily framed in terms of Li, the electrodepositions of Ag21 and Na22,23 in inorganic solid electrolytes were also extensively studied decades ago and many of the underlying core concepts are essentially transferable.

During Li plating, a homogenous layer-by-layer deposition between the anode and solid electrolyte (Fig. 3A) is desired for developing highly reversible solid-state batteries. The layer-bylayer electrodeposition of Li with bulk-type Li<sub>2</sub>S-P<sub>2</sub>S<sub>5</sub> glass<sup>24</sup> and Li<sub>7</sub>La<sub>3</sub>Zr<sub>2</sub>O<sub>12</sub><sup>25</sup> solid electrolytes has been demonstrated. Nevertheless, it is shown that this type of deposition can occur only at small current densities (i.e., low deposition rates). As the current density increases, the growth mechanism deviates significantly from the layer-by-layer mode and Li penetration through the solid electrolyte has been widely observed, causing cell shorting.<sup>26-28</sup> Borrowing from the concept in liquid-electrolyte lithium metal batteries,29 the formation of metallic Li inside the solid electrolyte is usually called dendritic growth, although the morphology of Li formed in solid electrolyte can be sharply different from the dendritic feature.<sup>19,26</sup> The conventional theories to explain the dendrite formation in organic electrolytes, based on transport limitation of the salt<sup>30</sup> and mechanics of electrolyte,<sup>31</sup> cannot be applied to solid-state Li metal cells.

Based on the previous study on Na plating in Na- $\beta$ -Alumina solid electrolytes,<sup>22</sup> the growth of metallic Li through the solid electrolyte is divided into two modes.<sup>19</sup> Mode I describes the Li growth at the interface of anode and solid electrolytes (Fig. 3B). This growth mode is caused primarily by current focusing (i.e., current hotspots) at the anode/electrolyte interface due to the existence of cracks, pores, impurities, and grain boundaries at the surface of solid electrolytes. The significantly enhanced current density at these local positions can easily exceed the self-diffusion limit of Li in the bulk, leading to the growth of Li toward the solid electrolyte through crack propagation.<sup>19</sup> On the other hand, Mode II describes the internal deposition of Li directly inside the solid electrolyte (Fig. 3C).<sup>28</sup> Internal deposition in solid electrolyte has also been experimentally



**FIG. 3.** *Electrodeposition from a solid-state electrolyte.* (*A*)

Electrodeposition of Li in solid-state lithium metal batteries: Layer-bylayer deposition under low current densities; (B) Directional growth of Li from the anode/electrolyte interface due to current focusing and Li flux imbalance; (C)Internal deposition of Li due to electronic conductivity of solid electrolyte.

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observed in the electrodeposition of Na and Ag.<sup>21,22</sup> The root cause of this growth mode is the residual electronic conduction in solid electrolytes.<sup>21,22</sup> While electronic transport in Li solid electrolytes has not been carefully studied,<sup>32</sup> inhomogeneous distribution of electronic conductivity inside solid electrolytes, possibly due to the surface electronic conduction of solid electrolyte,<sup>33</sup> reduction of solid electrolyte,<sup>22</sup> or enhanced conductivity at local defects such as grain boundaries,<sup>34</sup> can result in a gradient of transference number, leading to an overshot in the Li chemical potential as the driving force for direct formation of Li inside the solid electrolyte.<sup>23</sup>

It is still a matter of intense debate in the community as to which mode dominates the Li growth in inorganic solid electrolytes. Due to the much slower self-diffusion of Li versus the large localized current densities, Mode I growth has been considered as a faster process than Mode II. Nevertheless, one should note that these two modes cannot be considered as separate processes. The directional growth of Li through Mode I can increase the electronic conductivity of solid electrolytes and promote Mode II growth, while internal deposition of Li can extend crack formation and accelerate directional growth. The key information for determining which mode is dominant is to identify where the Li first nucleates at the first deposition, but this information has not been revealed due to challenges in characterizing the nucleation behavior of Li in solid electrolytes. Nevertheless, current understanding of the mechanism of electrodeposition of Li can provide some insights into the strategies to suppress Li penetration into solid electrolytes. To suppress Mode I growth, the surface of inorganic solid electrolytes should be carefully treated to remove cracks, pores, and other defects as much as possible prior to plating. Designing an additional interlayer, based on polymeric, metallic, or ceramic materials,<sup>35</sup> at the anode/electrolyte interface may also be helpful in achieving homogeneous current distribution, but these interlayers should sustain during repeated cycles. Another less-mentioned strategy is to improve the self-diffusion coefficient of Li in the substrate Li anode, for example by alloying or microstructure engineering.36 On the other hand, more careful studies of electronic transport in solid electrolytes are still needed to understand the voltage dependence, charge carrier, and root causes of electronic conductivity in solid electrolyte to eventually design strategies to suppress the internal deposition of Li.

#### Future Inquiries Toward the "Holy Grail" and Beyond

In addition to playing a critical role in anodes, electrodeposition has found important applications in enabling high-areal-capacity, thick batteries cathodes. A variety of transition metal oxides can be directly electroplated—at elevated temperatures from their corresponding molten salts—onto planar Al current collectors.<sup>37,38</sup> In particular, these electroplated electrodes manifest extraordinary rate capabilities at areal capacities (i.e., 20 mAh/cm<sup>2</sup>) that are a few times higher than normal commercial Li-ion electrodes. This type of dense electroplated cathode—possible because of the continuous ion and electron conduction network formed during electroplating—can be seamlessly integrated into solid state batteries. Future research might further enable electroplating of cathode materials that are less intrinsically electrically conductive.

The reverse reaction that occurs during battery discharge, electrodissolution, has been only rarely investigated in contemporary battery literature. A pioneering work by Shi et al. demonstrated that the electro-dissolution process plays an equally, if not more, important role in determining the morphological evolution and reversibility of Li metal anodes through mechanisms that have been underexplored to date.<sup>39</sup> While dendritic growth is mostly seen in mass transport controlled conditions during deposition, electro-stripping at the mass transport limit produces a bright, micro-finished surface.<sup>40</sup> More research is needed on this fundamental phenomenon, as well as on the interrelation between the stripping morphology and subsequent deposition morphology onto the stripped surface. This research will require borrowing past results in industries such as surface finishing

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that rely on electro-stripping and applying these in the context of batteries.

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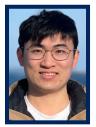


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