Long-Cycle-Life Calcium Battery with a High-Capacity Conversion Cathode Enabled by a Ca\(^{2+}\)/Li\(^+\) Hybrid Electrolyte

Zhen Meng,* Adam Reupert, Yushu Tang, Zhenyou Li, Guruprakash Karkera, Liping Wang, Ananyo Roy, Thomas Diemant, Maximilian Fichtner, and Zhirong Zhao-Karger*

ABSTRACT: Calcium (Ca) batteries represent an attractive option for electrochemical energy storage due to physicochemical and economic reasons. The standard reduction potential of Ca (−2.87 V) is close to Li and promises a wide voltage window for Ca full batteries, while the high abundance of Ca in the earth’s crust implies low material costs. However, the development of Ca batteries is currently hindered by technical issues such as the lack of compatible electrolytes for reversible Ca\(^{2+}\) plating/stripping and high-capacity cathodes with fast kinetics. Herein, we employed FeS\(_2\) as a conversion cathode material and combined it with a Li\(^+\)/Ca\(^{2+}\) hybrid electrolyte for Ca batteries. We demonstrate that Li\(^+\) ions ensured reversible Ca\(^{2+}\) plating/stripping on the Ca metal anode with a small overpotential. At the same time, they enable the conversion of FeS\(_2\), offering high discharge capacity. As a result, the Ca/FeS\(_2\) cell demonstrated an excellent long-term cycling performance with a high discharge capacity of 303 mAh g\(^{-1}\) over 200 cycles. Even though the practical application of such an approach is questionable due to the high quantity of electrolytes, we believe that our scientific findings still provide new directions for studying Ca batteries with long-term cycling.

KEYWORDS: FeS\(_2\) cathode, Ca anode, hybrid electrolyte, long-term cycling, Ca battery

1. INTRODUCTION

Multivalent-ion batteries have received increasing attention in recent years. In this context, calcium ion (Ca\(^{2+}\) ion)-based battery chemistry is one of the most promising candidates among these emerging technologies. The Ca/Ca\(^{2+}\) couple could theoretically deliver a capacity of 1340 mAh g\(^{-1}\) with a low standard reduction potential of −2.87 V, which is only 0.17 V difference from Li. Furthermore, Ca is the fifth most abundant element in the earth’s crust and is environmentally benign. Nevertheless, reversible Ca plating/stripping is still challenging due to the lack of compatible electrolytes. Recently reported fluoride-based electrolytes exhibit reversible Ca plating/stripping and high anodic stability. However, the CaF\(_2\) formation on the surface lowers the Coulombic efficiency and impedes Ca\(^{2+}\) migration.\(^{1−3}\) The electrolyte based on Ca(BH\(_4\))\(_2\) exhibited better Ca plating/stripping performance due to the absence of a passivation layer on the Ca surface. Although the oxidation stability is <3 V versus Ca/Ca\(^{2+}\), it is suitable to evaluate the cathode materials in this voltage window. Another hurdle toward rechargeable Ca batteries is the lack of proper cathode host materials. Different from the extensive success of intercalation cathodes in Li-ion batteries experimentally and theoretically, Ca-ion intercalation is challenging as the highly charged cation can induce polarization within the host environment.\(^{4}\) Organic materials and conversion cathodes based on sulfur have been investigated for Ca batteries very recently,\(^{5−7}\) which could circumvent the intrinsic drawbacks of intercalation chemistry and potentially offer high capacities.

To explore new cathode candidates and enhance the electrochemical performance of the multivalent batteries, the dual-salt electrolyte concept has been proposed, where Li\(^+\) or Na\(^+\) coexists with multivalent ions (Ca\(^{2+}\) and Mg\(^{2+}\)) in the electrolyte. These coexisting monovalent metal ions could primarily promote the redox reaction at the Ca or Mg anode by either being involved in the solid electrolyte interphase (SEI) formation\(^{8,9}\) or manipulating the solvation of multivalent ions.\(^{10}\) The usage of dual-salt electrolytes also provides wide options for cathodes. For instance, dual-salt electrolytes have been used for hybrid or dual-ion battery systems by combining a Li\(^+\)-ion intercalation cathode with a multivalent metal anode.\(^{11−16}\) Li\(^+\) ions could also mediate a polysulfide reaction, thus boosting the Mg–S and Ca–S battery performance.\(^{17,18}\) Other conversion cathodes, such as FeS\(_2\) and FeS, have been investigated with the Mg(BH\(_4\))\(_2\)/LiBH\(_4\)/tetrahydrofuran (THF) electrolyte,\(^{19}\) which combines the virtues of smooth
Mg plating/stripping and kinetically favorable Li-driven conversion of FeS<sub>2</sub>

Herein, an FeS<sub>2</sub>-based conversion cathode was employed for Ca batteries. FeS<sub>2</sub> has been investigated as a Li and Na storage material. Based on the redox reactions of Fe<sup>2+</sup>/Fe<sup>0</sup> and S<sup>2-</sup>/S<sup>2-</sup>, it offers a high theoretical capacity of 894 mAh g<sup>-1</sup>. However, our initial experiments indicated the absence of reversible conversion of FeS<sub>2</sub> in the Ca system with Ca[B(hfip)<sub>4</sub>]<sub>2</sub>/DME or Ca(BH<sub>4</sub>)<sub>2</sub>/THF single-salt electrolytes. In contrast, adding Li<sup>+</sup> ions by using the Ca(BH<sub>4</sub>)<sub>2</sub>/LiBH<sub>4</sub>/THF electrolyte could significantly boost the performance. The results proved that the Li<sup>+</sup> ion played a crucial role in enabling the reversible cycling performance of the cathode.

On the anode side, LiBH<sub>4</sub> may facilitate reversible Ca plating/stripping over long-term cycling by lowering the Ca<sup>2+</sup> ion desolvation energy. As a result, Ca batteries with long cycle life and stable capacity retention were demonstrated. It should be noted that a high electrolyte/cathode ratio is necessary to ensure the conversion (>22.5 μL/mg; see the Supporting Information, electrolyte-to-cathode ratio calculation), which will decrease the energy density of the cell. However, the positive effect of Li<sup>+</sup> on both the anode and cathode sides still provides a new direction for us to achieve a Ca battery with long cycle life.

2. EXPERIMENTAL SECTION

2.1. Preparation of FeS<sub>2</sub>. FeS<sub>2</sub> was synthesized according to previous literature. Briefly, Fe<sub>3</sub>O<sub>4</sub> and sulfur powder were mixed in a weight ratio of 1:1.5 and sealed into a quartz tube under vacuum, which was subsequently heated in a furnace with a ramp of 2 °C min<sup>-1</sup> at 600 °C and kept at this temperature for 2 h. After cooling down to room temperature, the powder was washed with CS<sub>2</sub> several times to remove the residual sulfur. The successful preparation of FeS<sub>2</sub> with a cubic structure was confirmed by X-ray diffraction (XRD) measurements (Figure S1). Furthermore, SEM images (Figure S2) showed that the particle size varied from 100 nm to micron.

2.2. Preparation of Electrolytes. THF was dried with 3 Å molecular sieves before use. Ca(BH<sub>4</sub>)<sub>2</sub>•2THF and LiBH<sub>4</sub> were used as received. Three electrolyte mixtures were prepared (1.5 M Ca(BH<sub>4</sub>)<sub>2</sub>/THF, 1.5 M Ca(BH<sub>4</sub>)<sub>2</sub> + 0.1 M LiBH<sub>4</sub>/THF, and 1.5 M LiBH<sub>4</sub> + 0.5 M Ca(BH<sub>4</sub>)<sub>2</sub>/THF) by adding appropriate amounts of Ca(BH<sub>4</sub>)<sub>2</sub>•2THF and LiBH<sub>4</sub> to THF (viz., 321 mg of Ca(BH<sub>4</sub>)<sub>2</sub>•2THF, 321 mg of Ca(BH<sub>4</sub>)<sub>2</sub>•2THF + 2.2 mg of LiBH<sub>4</sub>, and 107 mg of Ca(BH<sub>4</sub>)<sub>2</sub>•2THF + 33 mg of LiBH<sub>4</sub> into 1 mL of THF, respectively). After stirring overnight and waiting for 1 day, the supernatant was used as the electrolyte. It should be noted that the concentration values (1.5, 0.1, and 0.5 M) here are just used to improve the performance.
distinguish the electrolytes. They are not the real molarities of electrolytes since some insoluble residue was still visible in all cases after stirring overnight, and the electrolyte volume would change once the salt is added to the solvent. The exact values for these electrolytes were 1.22 M Ca(BH$_4$)$_2$/THF, 1.22 M Ca(BH$_4$)$_2$ + 0.082 M LiBH$_4$/THF, and 1.33 M LiBH$_4$ + 0.44 M Ca(BH$_4$)$_2$/THF, respectively (Supporting Information).

2.3. Electrochemical Measurement. For the FeS$_2$ electrodes, FeS$_2$ Super P, and PVDF were mixed in a weight ratio of 7:2:1 with NMP solvent. The slurry was dropped on the carbon paper, followed by drying under vacuum at 80 °C overnight. The FeS$_2$ loading was 1–1.5 mg cm$^{-2}$. For Ca electrodes, calcium shot was pressed into disks. Two pieces of glass fiber sheets (GF/C, Whatman) were used as separators for cell assembly. The electrochemical measurements of the FeS$_2$ electrodes were carried out with CR2032 coin cells with 80 μL of electrolyte. The electrolyte-to-cathode ratio is 113 μL/mg. Tests with symmetric cells were conducted with Swagelok cells with 60 μL of electrolyte. For the three-electrode test, a PAT-Cell (from EL-Cell) was used with 200 μL of electrolyte. The measurements were performed using a Bio-Logic VMP3 potentiostat and an Arbin battery cycling unit at room temperature.

2.4. Material Characterization. XRD measurement was carried out with a Bruker D8 ADVANCE XRD diffractometer with a Cu K$_\alpha$ source. A Zeiss LEO 1530 was used for scanning electron microscopy (SEM) analysis. Transmission electron microscopy (TEM) characterization was performed on a probe-corrected microscope Themis300 (Thermo Fisher Scientific) and a double aberration-corrected microscope ThemisZ (Thermo Fisher Scientific) at an acceleration voltage of 300 kV. The TEMs were equipped with a high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) detector and a Super-X EDX detector to acquire energy-dispersive X-ray spectroscopy (EDS) maps. The TEM specimen was prepared inside the glovebox by scratching the electrode material with a lacey carbon-coated gold grid. Afterward, the grid was mounted into a Gatan 648 vacuum transfer holder, which can be used to transfer the specimen into the TEM under Ar protection. The X-ray photoelectron spectra (XPS) were acquired on a Specs XPS system with a Phoibos energy analyzer using monochromatic Al K$_\alpha$ radiation (1486.6 eV), a take-off angle of 45°, and pass energies of 30 and 90 eV at the analyzer for detail and survey spectra, respectively. The samples were transferred under Ar from the glovebox to the XPS system to avoid contamination. CasaXPS was used for data analysis, using Shirley-type backgrounds and Gaussian–Lorentzian peak shapes.

3. RESULTS AND DISCUSSION

3.1. Electrochemical Performance of the FeS$_2$–Ca Cell. The electrochemical performance of the Ca/FeS$_2$ cells was investigated with three electrolytes, 1.5 M Ca(BH$_4$)$_2$/THF, 1.5 M Ca(BH$_4$)$_2$ + 0.1 M LiBH$_4$/THF, and 0.5 M Ca(BH$_4$)$_2$ + 1.5 M LiBH$_4$/THF. As shown in Figure 1a and Figure S3, in the 1.5 M Ca(BH$_4$)$_2$/THF electrolyte without LiBH$_4$ addition, FeS$_2$ delivered almost no capacity. Adding 0.1 M LiBH$_4$ into the 1.5 M Ca(BH$_4$)$_2$/THF electrolyte could trigger the electrochemical process between the FeS$_2$ cathode and the Ca anode, which was demonstrated by the increased capacity and a discharge plateau at around 1.1 V. Nevertheless, the discharge capacity was still limited to 25 mAh g$^{-1}$, suggesting a poor electrochemical activity of the Ca/FeS$_2$ system. Further optimization of the electrolyte concentration to 0.5 M Ca(BH$_4$)$_2$ + 1.5 M LiBH$_4$/THF significantly enhanced the electrochemical performance (Figure 1b,c). The Ca/FeS$_2$ cell exhibited a flat discharge plateau at around 1.3 V, delivering a high initial discharge capacity of 809 mAh g$^{-1}$. This is close to the theoretical capacity of the conversion from Fe$^{2+}$ to Fe$^{0}$ and S$^{2-}$ to S$^{0}$ reported in Li-ion batteries proving the high electrochemical activity of the Ca/FeS$_2$ cell. In the first cycle, the cell delivered a capacity of 496 mAh g$^{-1}$. The significant difference between the first discharge and charge capacities could imply that the electrochemical process of the FeS$_2$ in the first cycle is not reversible, which will be discussed later in this work. In the second cycle, the cell delivered a discharge capacity of 493 mAh g$^{-1}$ with a Coulombic efficiency of 98.3%. The average discharge potential was between 1.4 and 1.6 V, which is lower than the theoretical potential calculated (Supporting Information, calculation of the theoretical potential). After 200 cycles, a high discharge capacity of 303 mAh g$^{-1}$ with a Coulombic efficiency of 96.7% remained. The FeS$_2$–Ca cell with the 0.5 M Ca(BH$_4$)$_2$ + 1.5 M LiBH$_4$/THF electrolyte also exhibited an excellent rate performance (Figure 1d,e). It displayed discharge capacities of 474, 434, 375, and 317 mAh g$^{-1}$ at C/8, C/5, C/2, and 1C, respectively. When the C-rate gradually reverted to C/8, the discharge capacity was restored to 399 mAh g$^{-1}$.

3.2. FeS$_2$ Cathode Reaction Mechanism. The addition of LiBH$_4$ vastly improved the discharge capacity of the FeS$_2$–Ca cell in initial cycles, which may be due to the following two reasons: (1) the kinetic of the Li$^{+}$ ion is superior to that of the Ca$^{2+}$ ion, which is beneficial to the FeS$_2$ cathode reaction during charge/discharge, and (2) the LiBH$_4$ reduces the overpotential of the FeS$_2$–Ca cell significantly by decreasing

Figure 2. Voltage profiles of two-electrode (a, c, e) and three-electrode (b, d, f) FeS$_2$–Ca cells: (a, b) 1.5 M Ca(BH$_4$)$_2$/THF, (c, d) 1.5 M Ca(BH$_4$)$_2$ + 0.1 M LiBH$_4$/THF, and (e, f) 0.5 M Ca(BH$_4$)$_2$ + 1.5 M LiBH$_4$/THF.
the Ca anode plating/stripping overpotential, which could also increase the discharge capacity of the cell. To clarify which of the two mentioned hypotheses is dominant, three-electrode cell measurements were conducted. As shown in Figure 2, the overpotentials of the Ca anodes (blue curves) were lower than 0.3 V in these three electrolytes, which influenced the voltage profiles of the Ca/FeS₂ full cell negligibly. Instead, the voltage profiles of the full cells were dominated by the FeS₂ cathode side. In the 1.5 M Ca(BH₄)₂/THF electrolyte (Figure 2a, b), the FeS₂ cathode scarcely reacted during discharge, leading to a fast voltage drop and ultralow capacity. The discharge capacity increased with the addition of LiBH₄ (Figure 2c–f), proving that the Li⁺ ions were highly involved in the cathode discharge reaction. The three-electrode measurements proved that hypothesis 1, not hypothesis 2, is the reason for the high capacity of the FeS₂/Ca cell with 1.5 M LiBH₄ + 0.5 M Ca(BH₄)₂/THF in the initial cycles.

Ex situ XRD was used to clarify the cathode reaction mechanism in the 0.5 M Ca(BH₄)₂ + 1.5 M LiBH₄/THF electrolyte. As displayed in Figure 3, the FeS₂ peaks faded away during the first discharge, while two new broad peaks centered at 44.8° and 53.1° emerged. The signal at 44.8° can be indexed to Li₂S (220) and/or Fe (110). Since these two features overlap, precise identification is difficult. The other one at 53.1° corresponds to the Li₂S (311). The intensity of these two features was weakened during the first charge. However, signals of FeS₂ could not be observed even at the end of the charge (EOC), suggesting that the reaction in the first discharge may not be reversible. Afterward, the results indicated a reversible reaction in the following discharge as the two characteristic peaks at 44.8° and 53.1° gained intensity again. Taken together, the ex situ XRD demonstrated that the FeS₂ was converted to Li₂S at the end of discharge, which could not be oxidized to FeS₂ in the following charge, leading to a significant irreversible capacity loss. After the first discharge, the conversion became reversible.

High-resolution transmission electron microscopy (HRTEM) was employed to further reveal the reaction mechanism of the FeS₂ cathode. As shown in Figure 4, the selected area electron diffraction (SAED) of the cathode material at the end of the first discharge (EOD) displayed vague diffraction rings, which could be assigned to Li₂S and/or Fe (Figure 4a). The lattice fringes of these two products were also captured in images with a higher magnification (Figure 4b), proving the conversion reaction from FeS₂ to Li₂S and Fe. Nevertheless, it is still unclear whether the CaS was formed or not, given that Ca²⁺ and Li⁺ coexisted in the electrolyte and the d-spacing of CaS was similar to that of Li₂S (Figure S4). The elemental mapping of this sample is displayed in Figure S5. It demonstrated that the signals of sulfur and iron seem to be more or less evenly distributed across the whole nanoparticles. Calcium rather accumulates at the surface of the nanoparticles with a small atomic fraction of 5.1%, much lower than that of S (63.5%), implying that the calcium should be mainly from electrolyte residues. The HRTEM images and EDX results, combined with the three-electrode data in Figure 2, proved that Li⁺ was the dominant ion to drive the conversion reaction rather than Ca²⁺. Apart from Li₂S and Fe, the lattice fringes of Li₂FeS₄ were also seen in the HRTEM images of the discharged sample (Figure 4c). This was supposed to be the intermediate product from the FeS₂ reduction. Based on these results and previous reports, the reaction during charge and discharge is supposed to be the most widely accepted

Figure 3. Voltage profiles of the FeS₂−Ca cell with the 0.5 M Ca(BH₄)₂ + 1.5 M LiBH₄/THF electrolyte (a) and the corresponding ex situ XRD measurements (b).

Figure 4. TEM images of the samples at (a−c) EOD and (d−f) EOC. Insets of a and d: corresponding SAED patterns.
mechanism (Supporting Information, reaction mechanism part).21,26–28 Indeed, FeS$_2$ and Li$_2$FeS$_2$ became more substantial in the nanodomains at EOC (Figure 4d–f). No FeS$_{8/7}$ diffraction signal was detected, probably due to its low content.

The chemical state of FeS$_2$ cathodes in different charge/discharge states was also investigated in XPS measurements. Most importantly, the S 2p detail spectra provided more solid evidence for this conversion mechanism. As shown in Figure 5, a peak doublet was detected in the spectrum of the pristine FeS$_2$ at 162.9 and 164.1 eV, which can be assigned to the S$^{2-}$ of FeS$_2$.29–31 The peak doublet shifted to the lower binding energy of 160.1 and 161.3 eV at EOD, indicating the conversion from S$^{2-}$ to Li$_2$S.32,33 After recharging the cell to 2.2 V (EOC), the binding energy did not recover to the pristine state. Instead, the negligible binding energy change between EOD and EOC proved that most of the sulfur species were preserved as S$^{2-}$, consistent with HRTEM analysis.

3.3. Beneficial Effect of LiBH$_4$ on the Ca Anode.

After elucidating the reactions at the FeS$_2$ cathode and demonstrating the crucial role that Li$^+$ ions play here, we now turn to the Ca anode. Another impressive feature of the Ca/FeS$_2$ cell is its long cycle life, which indicates efficient Ca stripping/plating over long cycling. Concerning this, we evaluated the feasibility of the reversible Ca stripping/plating by galvanostatically cycling Ca/Ca symmetric cells in the three electrolytes (1.5 M Ca(BH$_4$)$_2$/THF, 1.5 M Ca(BH$_4$)$_2$ + 0.1 M LiBH$_4$/THF, and 0.5 M Ca(BH$_4$)$_2$ + 1.5 M LiBH$_4$/THF). As shown in Figure 6a,b, all three electrolytes exhibited moderate Ca plating/stripping overpotentials below 0.1 V over the first 20 h (except for the first activation cycle). Afterward, however, the voltage value of 1.5 M Ca(BH$_4$)$_2$/THF increased terribly, followed by severe fluctuations after 40 h, indicating a poor plating/stripping process. In sharp contrast, outstanding plating/stripping performance could be achieved in the electrolytes containing LiBH$_4$. The 1.5 M Ca(BH$_4$)$_2$ + 0.1 M LiBH$_4$/THF electrolyte delivered a plating/stripping overpotential lower than 0.1 V over 150 h. Remarkably, the 0.5 M Ca(BH$_4$)$_2$ + 1.5 M LiBH$_4$/THF electrolyte underwent over 200 h of charge/discharge cycles with low overpotential, which decreased gradually from 101 to 34 mV over the initial 70 h and then maintained constant over 200 h. It has been reported that the addition of 0.4 M LiBH$_4$ into the 0.4 M Ca(BH$_4$)$_2$/THF electrolyte could promote the plating/stripping reversibility of the Ca metal anode due to the enhanced desolvation kinetics of Ca$^{2+}$ ions in the electrolyte,10 which is also supposed to be
the determining factor for the superior performance of our 0.5 M Ca(BH$_4$)$_2$ + 1.5 M LiBH$_4$/THF electrolyte. To clarify whether the positive effect of the presence of LiBH$_4$ comes from the desolvation energy or the passive film formation, the XPS measurement of Ca electrodes after plating Ca under a current density of 0.1 mA cm$^{-2}$ was conducted, and the results are shown in Figure S6. The Ca 2p spectra with two peaks centered at 346.5 (Ca 2p1/2) and 350.0 (Ca 2p3/2) eV could be assigned to CaCO$_3$, suggesting that the CaCO$_3$ may be one of the components of the passivation layer. In the C1s spectra, three peaks centered at 284.8, 286.3, and 288.4 eV were assigned to C=C, C–O, and C=O bonds. These compounds may originate from THF decomposition or the adsorbents. The O 1s spectrum could be fitted into two peaks at 530.8 (C=O) and 532.5 (C–O) eV, which is in good agreement with the C spectrum. 34 No significant difference could be observed from the XPS spectra of these two electrode surfaces, indicating that the interfacial compositions or the passivation layers on the Ca electrodes are similar. This result is consistent with the previous report. 10 After Ca deposition, no XPS peak of Li could be detected on the electrode surface in the 0.5 M Ca(BH$_4$)$_2$ + 1.5 M LiBH$_4$/THF electrolyte, proving the lithium rather than calcium deposited on the anode during the electrochemical process under 0.1 mA cm$^{-2}$. The XRD pattern of the deposit on the Au working electrode was also recorded (Figure S7) to investigate the composition of the deposits. The Ca signals could be indexed, and no Li peak was observed, which is consistent with XPS data. It should be noted that we got this conclusion based on a current density of 0.1 mA cm$^{-2}$. The deposition behavior of this electrolyte under high current density may be different, given that higher current densities favor Li co-deposition for kinetic and mass transport reasons, which is currently under study. SEM and EDS analysis (Figure S8) shows that the deposited calcium takes on a round flower-like shape. The absence of dendritic calcium could reduce the possibility of penetrating through the separator and avoid a short circuit. However, it should be noted that the sample was exposed to air for a short time during being transferred to the SEM instrument. Calcium could react with oxygen during this period of time. That is why a certain amount of oxygen was also detected.

4. CONCLUSIONS

In summary, we report an attractive Ca battery with high capacity and long cycle life, composed of a conversion-type FeS$_2$ cathode and a metallic Ca metal anode. It combined the virtues of the remarkable capacity of FeS$_2$ and the ready availability of Ca due to its high abundance in the earth’s crust. The Ca(BH$_4$)$_2$ + LiBH$_4$/THF hybrid electrolyte ensured the kinetically favorable Li-driven conversion of FeS$_2$, thus bypassing the sluggish Ca$^{2+}$ ion diffusion in the cathode lattice. Simultaneously, it enhanced the desolvation kinetics of Ca$^{2+}$ ions, resulting in excellent reversibility with a small overpotential at the Ca anode over long-term cycling. Consequently, the FeS$_2$–Ca battery exhibited a high discharge capacity of 303 mAh g$^{-1}$ with a Coulombic efficiency of 96.7% over 200 cycles, which is superior to other batteries using a dual-salt electrolyte and a calcium metal anode (Table S7). Compared with some other conversion cathode/Ca cells, our concept could achieve long cycle life with commercially available salt easily. Further investigation about this concept will focus on how to reduce the electrolyte/Li$^+$ amount to make it more practical.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsami.2c11337.

SEM, EDS, measurements of FeS$_2$ materials, electrochemical performance, lattice parameter of Fe, Li, S, CaS, and Li$_2$FeS$_4$, and XRD of Ca depositions (PDF)

AUTHOR INFORMATION

Corresponding Authors

Zhen Meng — Helmholtz Institute Ulm (HIU) 
Electrochemical Energy Storage, Ulm D-89081 Baden-Württemberg, Germany; orcid.org/0000-0001-5756-9159; Email: zhen.meng@partner.kit.edu

Zhirong Zhao-Karger — Helmholtz Institute Ulm (HIU) 
Electrochemical Energy Storage, Ulm D-89081 Baden-Württemberg, Germany; orcid.org/0000-0002-7233-9818; Email: zhirong.zhao-karger@kit.edu

Authors

Adam Reupert — Helmholtz Institute Ulm (HIU) 
Electrochemical Energy Storage, Ulm D-89081 Baden-Württemberg, Germany

Yushu Tang — Institute of Nanotechnology, Karlsruhe Institute of Technology (KIT), Eggenstein-Leopoldshafen D-76344 Baden-Württemberg, Germany

Zhenyou Li — Helmholtz Institute Ulm (HIU) 
Electrochemical Energy Storage, Ulm D-89081 Baden-Württemberg, Germany; orcid.org/0000-0001-9624-2124

Guruprakash Karkera — Helmholtz Institute Ulm (HIU) 
Electrochemical Energy Storage, Ulm D-89081 Baden-Württemberg, Germany

Liping Wang — Helmholtz Institute Ulm (HIU) 
Electrochemical Energy Storage, Ulm D-89081 Baden-Württemberg, Germany; orcid.org/0000-0002-4113-2208

Ananyo Roy — Helmholtz Institute Ulm (HIU) 
Electrochemical Energy Storage, Ulm D-89081 Baden-Württemberg, Germany

Thomas Diemant — Helmholtz Institute Ulm (HIU) 
Electrochemical Energy Storage, Ulm D-89081 Baden-Württemberg, Germany

Maximilian Fichtner — Helmholtz Institute Ulm (HIU) 
Electrochemical Energy Storage, Ulm D-89081 Baden-Württemberg, Germany; Institute of Nanotechnology, Karlsruhe Institute of Technology (KIT), Eggenstein-Leopoldshafen D-76344 Baden-Württemberg, Germany; orcid.org/0000-0002-7127-1823

Complete contact information is available at: https://pubs.acs.org/doi/10.1021/acsami.2c11337

Notes

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