

Lithium–Sulfur Batteries: 3D Printed Tools and Assembly Techniques for Repeatable Lab-Scale Coin Cell Manufacturing

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Daryl Miranda, Tosif Aliyev, Lamija Kovacevic, Sivapriya Voleti, Jaydie Lee, Kathleen Meehan, Monica C. So, and Philip T. Dirlam*



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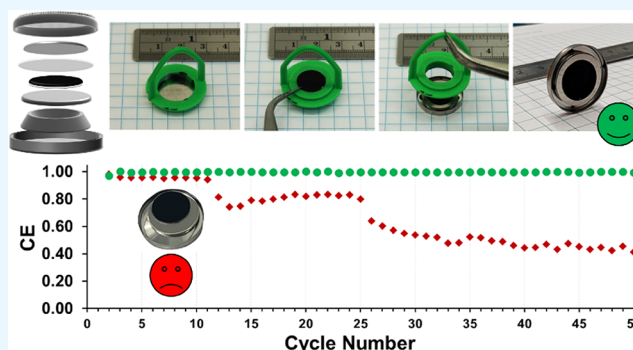


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ABSTRACT: The coin cell form factor has become a standard in energy storage research owing to its relatively low cost, low quantity of requisite electroactive material, fast experimental turnover, and capability to generate publishable quality data. Coin cell assembly at the lab-scale requires precise manual alignment of the many internal components of a cell to yield a properly functioning device. Truly repeatable device assembly presents a significant challenge to the acquisition of reliable data. In this report, we present a three-dimensional (3D) printable design for a coin cell alignment device and describe techniques for a generalized workflow to achieve reproducible coin cell assembly of lithium-based batteries. Standardized assembly techniques using readily accessible, purpose-built tools were demonstrated for lab-scale production of lithium–sulfur (Li–S) coin cells. Li–S batteries fabricated using the alignment device as well as conductive adhesive incorporated workflows afforded coin cells with up to 35% reduction of assembly times, comparable or better functional cell yields, and improved electrochemical device performance with more consistent and up to 150 mAh/g higher average discharge capacities coupled with Coulombic efficiency increases of up to 2.0% over manually aligned cell assembly techniques.



1. INTRODUCTION

In situ and operando testing is a necessity in the development of materials for new energy storage chemistries. Seldom is the theoretically maximal performance of a material in a battery realizable in experimentation. Inefficient redox chemistry, device impedance, cell age, or any of a number of matrix effects in a finished cell, could completely change the resultant electrochemical capabilities of a battery.^{1,2} In situ and operando testing serves to elucidate the state of the cell in that respect. Consideration then must be made toward which cell form factor is most suitable for test purposes.^{3,4} Pouch cells, prismatic cells, and cylindrical cells have achieved broad adoption in commercial and industrial applications as standard formats.^{5–9} But in comparison to coin cells, these cell designs are less amicable to laboratory scale experimentation with precious or exploratory electroactive materials, and can require substantial specialized equipment for processing and device fabrication.¹⁰ The relatively small amount of requisite active material necessary for a finished coin cell, coupled with the relatively low cost of cell components and processing equipment needed for lab-scale coin cell fabrication, presents an attractive test medium for research in the field.¹¹

There are, however, challenges inherent in coin cell fabrication that limit the reliability, and reproducibility of electrochemical device performance data generated.¹² One of the notable problems with lab-scale coin cell manufacturing stems from the requirement to assemble the case, spring, spacer, cathode, separator, and anode into a precisely concentrically aligned stack for the final cell to function properly.^{13,14} The sequential addition of six or more components creates a compounding variation in the final configuration of the coin cell as addition of each subsequent component increases the probability for misalignment of the overall assembly, which ultimately leads to distorted cell performance.¹⁵ Furthermore, many next-generation battery chemistries that are under investigation utilize metallic lithium or other reactive species,^{16–19} which require handling in an

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inert atmosphere necessitating the use of a glovebox. The reduced dexterity imparted in a glovebox exacerbates challenges of achieving submillimeter precision when manually assembling cells increasing the likelihood for misalignment of cell components. Kim et al. describe the results of electrode misalignment in Lithium-ion coin cells, and demonstrate a significant loss in Coulombic efficiency and substantial capacity fade as a result.¹³ This shows that data collected regarding electroactive material performance for a cell becomes difficult to parse from resulting deficiencies in component alignment without significant, and thorough, post-mortem analysis. Metrics that instill confidence in the functionality of a cell such as high Coulombic efficiency and capacity retention must then be prioritized when assessing new electrochemical systems in coin cells.²⁰ In other cases, improper alignment of a separator may result in direct contact of electrodes, yielding a nonfunctional cell.²¹ Thus, it is imperative for researchers to take care in the assembly process. Traditionally, this entails repeated practice assembling multiple cells until reliable, reproducible cells can be made; Luc et al. highlight this when describing the influence of the individual cell assembler's experience on the performance of coin cells.²² This presents a significant learning curve before meaningful work can be produced by inexperienced assemblers (e.g., undergraduate researchers) and introduces notable variation with coin cells produced among different researchers and between laboratories. Significant work has been done to make the assembly process more repeatable. Tools such as vacuum pens have been used by some researchers to allow for easier manipulation of small coin cell components.²³ But this tool can only remediate some of the difficulties in assembly, and the highly reactive nature of lithium metal limits what materials can be used for direct contact. Fully automated assembly devices have been developed for high throughput coin cell production, but these solutions are less feasible for lab-scale experimental chemistries and require comparatively complex assembly of the device.^{24–26}

Herein, we report the development of readily accessible three-dimensional (3D) printable tools and a unified workflow for repeatable production of coin cells with a significantly lower barrier for entry for researchers new to coin cell assembly. We demonstrate that the coin cells produced using the alignment device-assisted assembly methods described enable a high degree of consistency in the alignment of the myriad internal components compared to unassisted manual assembly, with improved assembly times, specific charge/discharge capacities, Coulombic efficiencies, and comparable functional cell yield. The improved reproducibility of battery test data and device performance is exemplified with lithium–sulfur (Li–S) coin cell batteries in this study.

2. RESULTS AND DISCUSSION

2.1. General Coin Cell Assembly Process. Li–S coin cells were assembled by dividing the seven solid cell components into two groups. A top assembly (anode case, lithium metal anode, and separator) and a bottom assembly (cathode, spacer, spring, and cathode case) were prepared separately and then joined together after the introduction of electrolyte solution onto the cathode (Figure 1). This order of assembly was chosen to both minimize the amount of time the electrolyte solution is openly exposed prior to capping the overall assembly and reduce the frequency of individual component placement events and the associated risk of

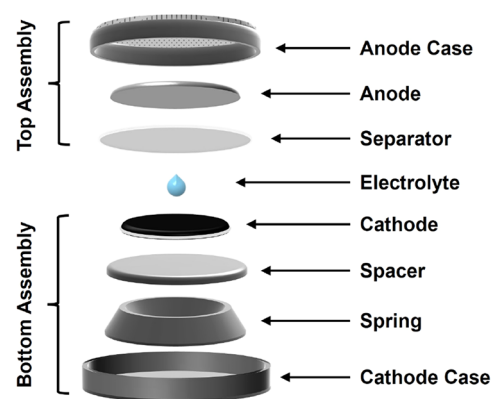


Figure 1. Schematic representation of the components of a coin cell with a relative position in the stacked assembly.

misalignment. The volume of electrolyte solution for Li–S batteries must be minimized to ensure adequate device performance (e.g., 10 μL per coin cell) and commonly a mixture of 1,3-dioxolane and 1,2-dimethoxyethane is employed as the solvent system, which is notably volatile. Delay in completing the assembly with placement of the anode cap after depositing the electrolyte solution allows for evaporation of the minute volume of electrolyte solvent significantly changing the amount and concentration of electrolyte in the cell. Furthermore, the coin cell crimpers commonly used in research lab settings operate with the cathode case on the bottom. This assembly order precludes the need to invert the cell before it is placed in the crimper, avoiding disruption of the component arrangement before the cell is compressed and sealed.

The process for constructing the top assembly is depicted in Figure 2. The top assembly consisted of the anode cap, lithium foil anode, and a Celgard separator. Anode caps were first modified by spot-welding a nickel tab to the interior of the anode cap (see Figure S1). This allowed the soft lithium foil to be manually pressed into the cap using a smooth, flat-ended, nonreactive, polyethylene rod. The nickel ribbon held the lithium foil in place throughout the assembly process. To improve reproducibility, a watch press was modified by replacing the head with a high molecular weight polyethylene tip. After the lithium was affixed to the cap, the separator was adhered to the lithium foil surface via static charge imparted to the separator via the triboelectric effect by shaking it in a plastic container.

The bottom assembly of the coin cell consisted of the cathode cap, spring, spacer, cathode, and electrolyte. Considerable care and practice were required to align these components concentrically, as they are stacked in the order shown in Figure 1. After the deposition of the electrolyte, the top assembly held with a suction cup was lowered atop the bottom assembly.

2.2. Alignment Device Design Rationale and Implementation. Given the workflow described above, each component manually added to the bottom assembly stack increases the frequency and extent of misalignment. A device was designed to retain the alignment of these components during the assembly process. Digital modeling and 3D printing allowed for fast device prototyping. With ready access to 3D printing services becoming common (e.g., public libraries, makerspaces at academic institutions), the low material cost

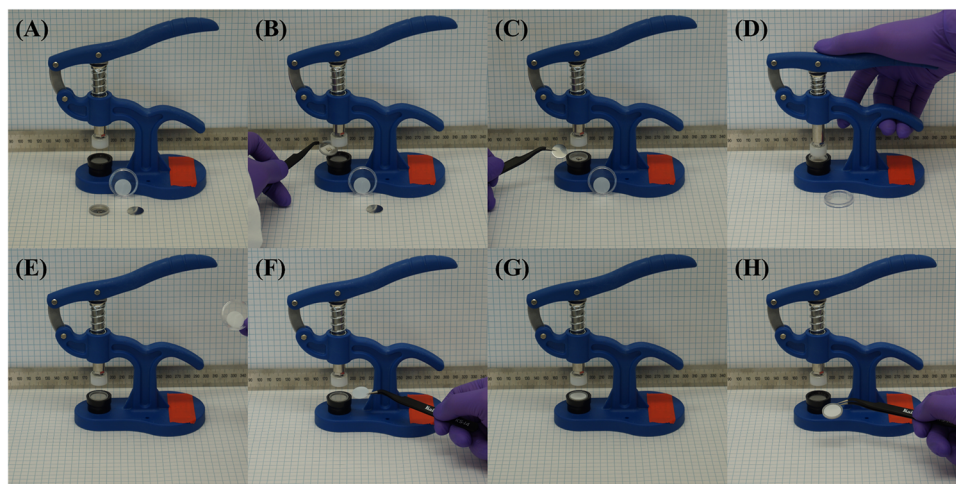


Figure 2. Depiction of the process for preparing the top assembly with the use of the lithium press tool. (A) Example of the components used in the top case assembly, lithium press tool, modified anode case, anode material, and separator in a plastic case. (B) Placement of the anode case in the Li-press followed by (C) placing the metal anode into the anode case and (D) pressing the anode to affix it to the case. The separator is then prepared by (E) imparting a static charge by shaking it in the plastic case, (F) after which it is removed, and (G) allowed to adhere to the anode material via static cling action affording (H) completed top assembly removed from the press tool to be used in the full cell assembly.

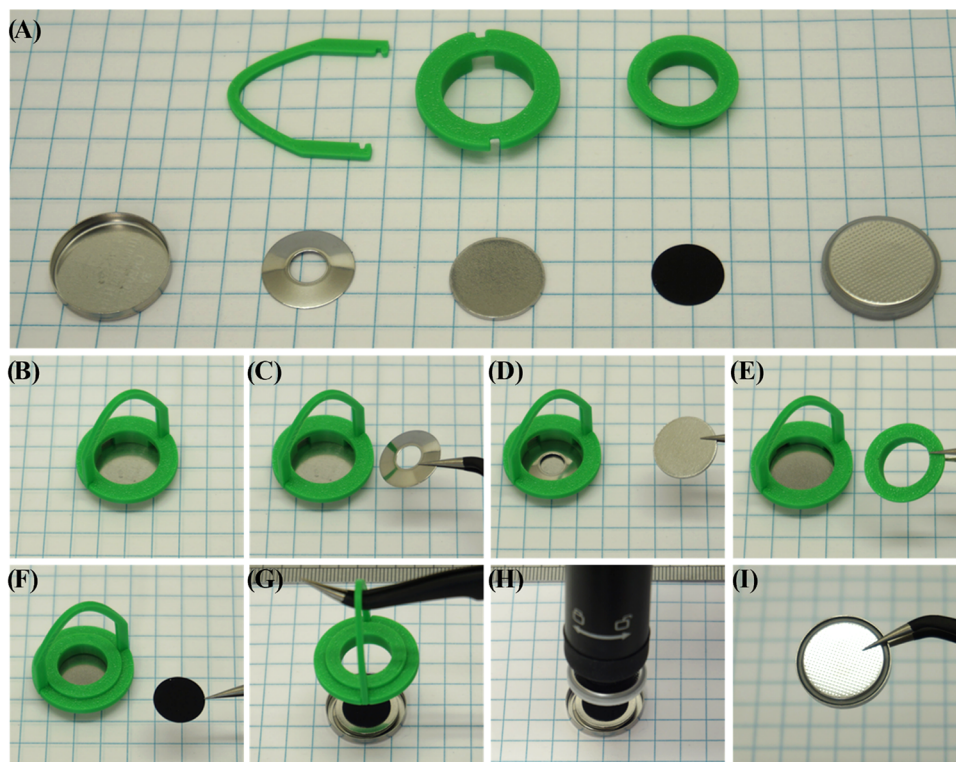


Figure 3. Depiction of CR2032 coin cell assembly using the two-piece alignment device and suction cup. (A) Components used in the coin cell assembly process, including (top row) two-piece alignment device with handle and (bottom row, left to right) cathode case, spring, spacer, cathode, and top assembly. The assembly process was completed with (B) installing the alignment tool in the positive case followed by inserting the (C) spring, (D) spacer, (E) cathode aligner insert, and (F) cathode. (G) Removal of the alignment device using the attached handle, followed by dispensing electrolyte and (H) placement of the top assembly using a suction pen to yield a (I) well-aligned coin cell assembly ready to be crimped.

and ready access also present an attractive solution versus complex fully automated assembly methods.

The alignment device was designed with the goal of filling the space between the case, spring, spacer, and cathode during the alignment process. The device could then be removed, allowing for the deposition of the electrolyte and placement of the top assembly, yielding a finished battery assembly ready for crimping (Figure 3). To account for the larger diameter of the

spacer and spring relative to the cathode, concentric alignment for the components was achieved using a nested two-insert design. The first insert aligned the spring and spacer, while the second smaller insert aligned the cathode on the surface of the spacer.

2.3. Conductive Adhesive-Assisted Assembly. Coin cell assembly using conductive adhesive was investigated to determine whether components could be effectively immobi-

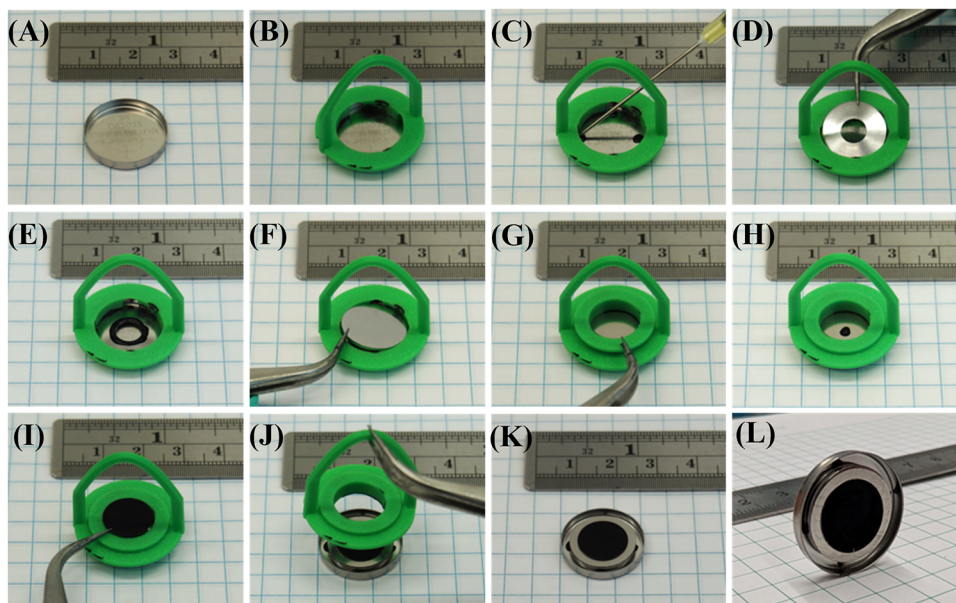


Figure 4. Demonstration of conductive adhesive-assisted assembly of a CR2032 coin cell using the two part alignment device depicting order of operations with (A) the cathode case, (B) alignment tool insertion, (C) application of conductive adhesive in the notches of the alignment device, (D) addition of spring, (E) application of conductive adhesive applied along the top small diameter washer spring surface, (F) addition of the spacer, (G) installation of the cathode aligner insert, (H) conductive adhesive applied to the spacer surface, (I) placement of the cathode, (J) removal of the alignment device followed by (K) brief rest allowing adhesive to set/dry yielding (L) the completed bottom assembly with alignment secured by conductive adhesive.

lized during the cell assembly process, further enhancing reproducibility and enabling preparation of the bottom assembly outside of the glovebox. Adhesive was applied between the cathode cap and spring, between the spring and the spacer, and between the spacer and the cathode (Figure 4). The alignment device was designed with notches to guide placement of the adhesive paint for proper contact with the aligned spring and to prevent adhering the alignment tool to the cell case. After the adhesive was dry, the now secured bottom assembly was taken into the glovebox and the full cell was completed with the anode side top assembly. Coin cells without electroactive cathode material were later tested using electrochemical impedance spectroscopy (EIS) to determine if the application of conductive adhesive had any effect on the impedance of the cells. EIS revealed low variation in the resistance attributed to the cell case, components, and electrolyte, determined from the high frequency regime (Figure S3). Cells assembled without conductive adhesive produced resistance values of 0.93 and 1.42 Ω , while cells assembled using the conductive adhesive exhibited comparable resistance values of 1.10 and 1.13 Ω , which indicated that the effect of the adhesive is negligible.

2.4. Evaluation of Successful Alignment. Visual inspection was used to determine the efficacy of the alignment device with regard to alignment determinations. AutoCAD was used to visually determine the precision of the alignment from photographs taken of the bottom assembly components. This was done for a bottom assembly stacked without the alignment device, with the use of the alignment device and with the alignment device and glue. The center point of the cathode case, cathode, spacer, and spring were measured. The distances between the center points of the bottom assembly components and the cathode case were then determined to quantify the accuracy of the concentric alignment. The variation in concentricity for cells assembled without the alignment device

was notable. Components could range from 0.05 to 1.50 mm off center relative to the cathode case. Using the alignment device, this variation was significantly smaller, with values ranging from 0.18 mm to 0.36 mm (Figure 5). There was no significant impact of the application of the conductive adhesive on component alignment in comparison to alignment assisted with just the device.

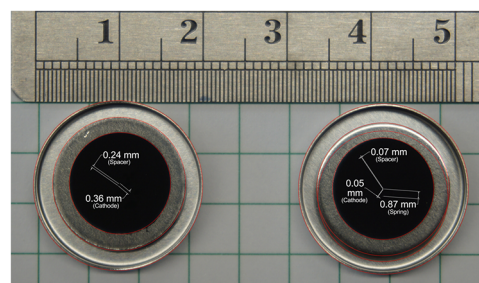


Figure 5. Representative bottom assemblies after assisted alignment using the alignment tool with adhesive (Left) and without adhesive (Right). The distance between the parallel lines corresponds to the deviation of the component center point to the center point of the cathode case.

Uncrimping cells assembled using assisted and adhesive-assisted assembly methods revealed no notable effects on the cell in comparison with manually assembled cells. The impression left on the separator by the cathode showed that the lithium foil had completely eclipsed the cathode surface. The adhesive, when used sparsely in cells, did not come into contact with the electrodes or separator.

2.5. Effect of the Modified Workflow on Speed and Reliability of Assembly. The effect on the speed of assembly using the alignment device was notable. Manually assembled batteries averaged 4.5 min per cell, while assisted cell

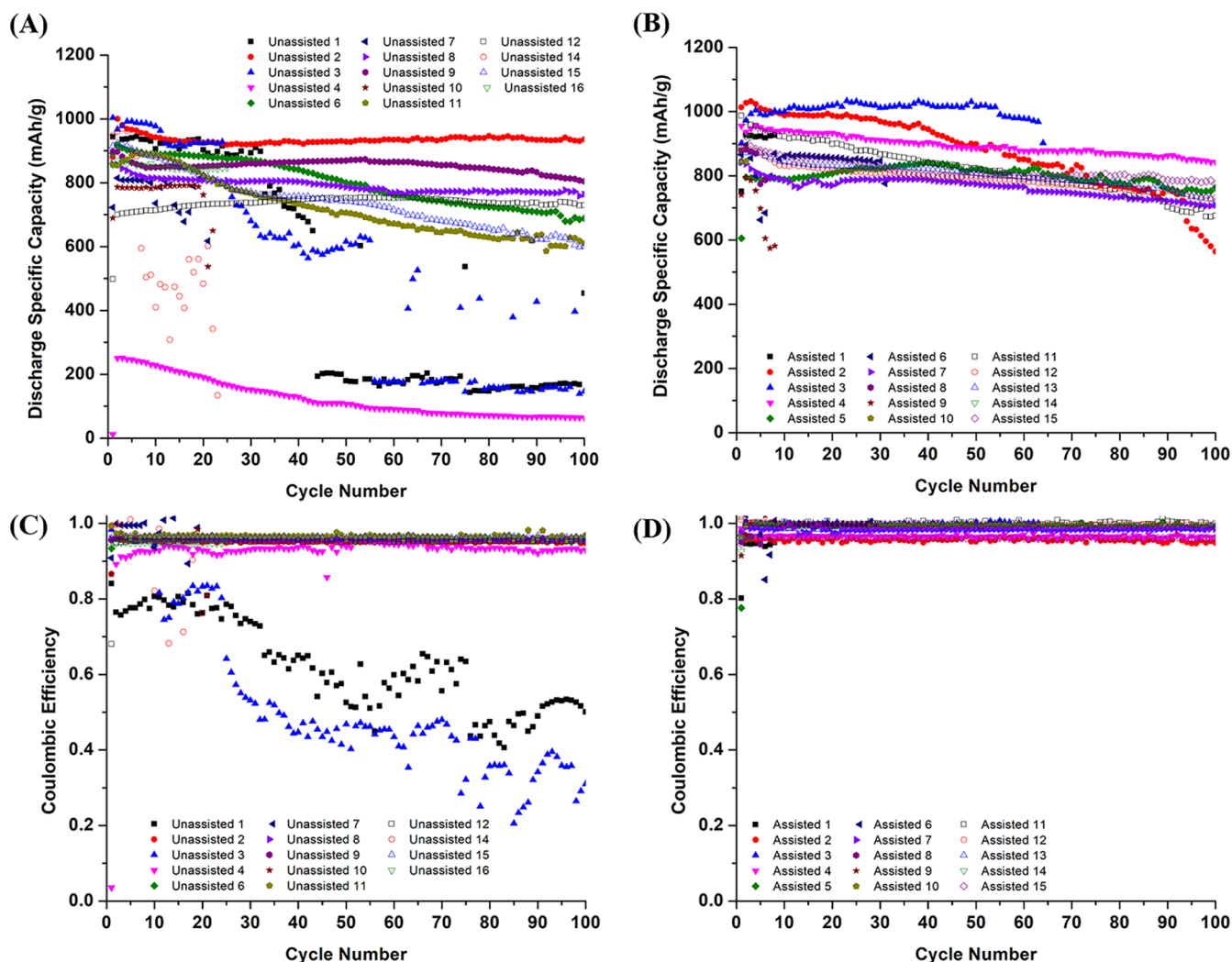


Figure 6. Battery cycling data plots showing (A, B) the specific discharge capacities and (C, D) Coulombic efficiencies versus cycle number of all viable Li–S coin cells fabricated with (A, C) manual alignment (unassisted) and (B, D) alignment device-assisted assembly. Cells were cycled at a rate of 0.75 C from 1.7 to 2.6 V versus Li/Li⁺ for up to 100 cycles.

construction methods averaged 3.5 min per cell corresponding to 20% improvement in assembly time cost. During a manual assembly, a significant amount of time is dedicated to repositioning the components in the bottom assembly. Repeated manipulation of the cathode in this respect introduces an increased risk of damage to the cathode surface. Pitting in the surface or total delamination of portions of the cathode may result. Using the alignment device substantially minimizes this risk, owing to the drop-in nature of the design. Use of the conductive adhesive further trivializes the alignment process as it can be completed outside of the glovebox, with the average assembly times constituting 1.7 min on the lab bench and 1.2 min in the glovebox (2.9 min total per cell) corresponding to over 35% improvement in time cost. Bottom assemblies with even small portions of adhesive were resilient to significant shocks, allowing for alignment to be retained throughout movement from the lab bench to the antechamber and to the interior of the glovebox. This persisted through cell construction, where the application of electrolyte and top assembly was fast and free of noticeable misalignments.

2.6. Effect of Assisted Alignment on Battery Performance. To determine postassembly if a cell was viable, the open circuit potential of the cell was measured shortly after assembly

during the initial rest period. If an open circuit potential within the range of 2.30–2.45 V was measured, the battery was determined to be viable. Cells were then galvanostatically charged/discharged at a current density corresponding to 0.75C between 2.6 and 1.7 V for up to 100 cycles. Specific discharge capacity, capacity fade, and overall Coulombic efficiency were used as the key metrics in determining overall cell performance.

Cycle life studies show the functional yield of cells using unassisted and assisted assembly procedures (Figure 6). Unassisted assembly showed a markedly wide range of discharge specific capacities and Coulombic efficiencies. Alignment device-assisted assembly procedures displayed more uniform discharge capacities and more uniform Coulombic efficiencies, with most cells retaining greater than 95% Coulombic efficiency throughout their functional lifetimes. Comparisons between the unassisted and assisted assembly data sets, with respect to the number of cells that cycled for the full 100-cycle test period, showed that 7 of 16 unassisted cells and 8 of 16 assisted cells retained a specific capacity of >500 mAh/g.

Adhesive-assisted cells tested using identical parameters displayed a wider range of discharge capacities and Coulombic

efficiencies (Figure S6.) Though the functional yield was improved to 11 of 16 assembled cells remaining functional over the 100-cycle test regimen. The influence of assembly technique on Li–S coin cell performance was evaluated by comparing discharge capacity and Coulombic efficiency across manually assembled, tool-assisted, and adhesive-assisted cells. A Welch's one-way ANOVA revealed a significant effect of assembly method on the average discharge capacity ($F(2, \approx 37) = 5.26, p = 0.009$). Games–Howell posthoc analysis showed that assisted assembly yielded significantly higher mean discharge capacities than both unassisted ($p = 0.008$) and adhesive-assisted ($p = 0.033$) assemblies, while no significant difference was observed between the latter two ($p = 0.12$). A comparison of Coulombic efficiencies of the three cell assembly types utilizing trimmed means was also carried out (Figure S7). Unassisted and assisted adhesive cells showed overall fluctuations of Coulombic efficiencies ranging less than 0.5%, whereas the deviation of trimmed mean values for cells built using conductive adhesive showed a notably larger range of 2.5%. In both the assisted and adhesive-assisted cell assemblies, the mean Coulombic efficiency was improved by more than 2.0% and 0.5%, respectively, over the unassisted assembly workflow.

3. CONCLUSIONS

During the coin cell assembly processes commonly carried out in a laboratory setting, significant variation in cell component alignment can manifest during manual assembly causing detrimental effects on device performance and inconsistent or erroneous data, especially when the assembly is being performed by various researchers with different levels of experience. The resulting poor yield of usable experimental data is a significant drain on testing time and experimental resources. The work described in this report is aimed at remediating this. We have developed a set of purpose-built 3D-printable tools and techniques with the aim of generating a repeatable procedure for coin cell construction, specifically addressing the issues of concentric alignment of components before cell crimping. Li–S cells built using this workflow were able to demonstrate a low variation in visually measured concentricity. The application of conductive adhesive was also tested as an immobilizing agent for the cell construction process. Electrochemical impedance spectroscopy was used to show that the adhesive demonstrated no measurable effect on device resistance and could even yield better-performing cells. Average assembly times for alignment assisted and adhesive-applied cells under inert atmospheric conditions were also improved over manually aligned cells. Cycle life studies were able to demonstrate overall improvements in specific charge/discharge capacities as well as Coulombic efficiencies of cells built using this improved methodology over coin cells built using manual alignment methods. An expanded study conducted with cell assemblers of varying experience would be prudent to improve the confidence in the findings described here. Future work should also include validating the workflow described here for other battery chemistries in addition to Li–S to ensure that this approach is broadly applicable across lab-scale battery research using coin cells.

■ ASSOCIATED CONTENT

SI Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.5c07669>.

Experimental details (materials, instrumentation, equipment, procedures, and directions for accessing the 3D models of the tools developed in this study), supplementary electrochemical impedance spectroscopy and battery cycling data for coin cells prepared with adhesive-assisted coin cell assembly procedure (PDF)

■ AUTHOR INFORMATION

Corresponding Author

Philip T. Dirlam – Department of Chemistry, San Jose State University, San Jose, California 95112-0101, United States; orcid.org/0000-0003-1900-6384; Email: philip.dirlam@sjsu.edu

Authors

Daryl Miranda – Department of Chemistry, San Jose State University, San Jose, California 95112-0101, United States;

orcid.org/0009-0002-3026-6902

Tosif Aliyev – Department of Chemistry, San Jose State University, San Jose, California 95112-0101, United States

Lamija Kovacevic – Department of Chemistry, San Jose State University, San Jose, California 95112-0101, United States

Sivapriya Voleti – Department of Chemistry, San Jose State University, San Jose, California 95112-0101, United States

Jaydie Lee – College of Natural Sciences, California State University Chico, Chico, California 95929-0210, United States

Kathleen Meehan – Electrical and Computer Engineering Department, California State University Chico, Chico, California 95929-0210, United States

Monica C. So – Chemistry and Biochemistry Department, California State University Chico, Chico, California 95929-0210, United States; orcid.org/0000-0002-9044-4806

Complete contact information is available at:

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Notes

The authors declare no competing financial interest.

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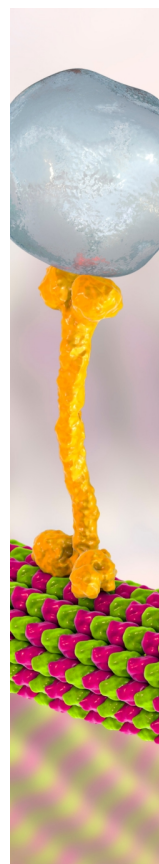
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