Tin Networked Electrode Providing Enhanced Volumetric Capacity and Pressureless Operation for All-Solid-State Li-Ion Batteries

Justin M. Whiteley,a Ji Woo Kim,a,c Chan Soon Kang,a Jong Soo Cho,a Kyu Hwan Oh,b,z and Se-Hee Leea,z

aDepartment of Mechanical Engineering, University of Colorado at Boulder, Boulder, Colorado 80300-0427, USA
bDepartment of Materials Science and Engineering, Seoul National University, Seoul, 151-742, Korea

cPresent address: Battery Research & Development Division, LG Chem Research

In 1997, Fuji announced a tin (Sn)-based amorphous composite oxide material for commercial Li-ion batteries.1 Ensuing anode deployments include the Sony developed Sn-based compound (Sn-Co-C) in 2005.2 Although these were the first commercial deployments of Sn-based anodes, Sn has been extensively studied for decades as a candidate in rechargeable Li-ion batteries because of its substantial lithium storage capabilities and quicker charging times.3 Despite the theoretical capacity of Sn being lower than the currently highlighted silicon (Si) anode, Sn has exceptionally appealing features: high gravimetric and volumetric capacity (959 mAh g−1 and 2,476 mAh mL−1 for 4.25 Li-ions),4 excellent electrical conductivity (9.17 × 107 S m−1), and room temperature Li-ion diffusivity (5.9 × 10−7 cm2 s−1 of Li1.5Sn).5 The commercialized Sn-Co-C anode by Sony provides a significant capacity advantage over the currently utilized graphite anode material (372 mAh g−1). However, wide use of the Sn-Co-C anode has been limited due to the high cost and environmental concerns about cobalt. Iron and nickel have been introduced as replacements for cobalt forming amorphous Sn-Fe and Sn-Ni with similar electrochemical properties to the Sn-Co alloy.6–10 Despite the low cost and high capacity of the Sn-Fe and Sn-Ni anodes, poor cycling stability and coulombic efficiency (CE) hinder their practical use in Li-ion batteries. These drawbacks mainly result from the enormous volume change of Sn (~255% when 4.25 Li-ion inserted),4,11 leading to a loss of electric contact, pulverization, and cracking.1,2 Therefore, controlling the microstructure of the expandable active material during lithiation/delithiation processes is a key point to realize a high energy-dense Li-ion battery using Sn-based anode materials.

Recently, Molina Piper et al. reported the effect of compressive stress on the electrochemical performance of a Si anode in an all-solid-state Li-ion cell, which similarly suffers from pulverization due to immense volume changes.13 By applying external compressive stress to the silicon/solid-state electrolyte (SSE) composite anode, free volume expansion of Si as well as solid-solid interfaces between the active material, SSE, and conductive additive were effectively controlled, thereby significantly reducing capacity fade. Generally, interfacial impedance in solid-state cells is much higher than that of conventional liquid electrolyte cell because the junction is limited to the small contact area between SSE and active material particles.14 Therefore, achieving and preserving absolute interfaces between SSE and active material during the cycling process will be one of the most critical points for highly reversible all-solid-state Li-ion batteries.

In this study, we aim to demonstrate improved cycling performance and reduced stack pressure of a high capacity Sn metal-based anode in an all-solid-state Li-ion battery. As Sn is one of the most ductile and malleable metals, we develop an interpenetrating network of electrically conductive Sn with ionically conductive SSE. The absence of conductive additive, such as carbon black, allows for enhanced cycle life as there is no concern for active material segregation. The composite anode delivers near theoretical capacity on the first cycle with over 99.9% coulombic efficiency,15–17 excellent electrical conductivity (9.17 × 107 S m−1),18 and high gravimetric and volumetric capacity (959 mAh g−1 and 2,476 mAh mL−1 for 4.25 Li-ions).19 Therefore, to ensure sustained particle-particle contact, external pressure is necessary in the range of a few MPa. This isn’t a realistic possibility for a commercial solid-state design. Thus, future work on solid-state architectures should focus on microstructural design of the electrode to address this issue.

To this resolve, exploration of Sn-based anodes in the solid-state configuration has been rather limited. To create the inherently conductive network, studies have focused on integrating Sn into glasses or crystals that act as conversion materials.15–17 These solutions usually result in very low amounts of Sn active material mass loading and still require significant amounts of conductive additive, essentially nullifying the attractive volumetric capacity from Sn. Additionally, almost all solid-state studies do not address the fact that these composite anodes, or even cathodes, require large amounts of external pressure for proper operation.18 Usage of a polymeric binder in solid electrolyte particles.

Experimental

All procedures outlined were conducted in a dry argon environment. To assemble test cells, a 200 mg glass solid electrolyte pellet separator was first pressed at 1 metric ton inside a polyetheretherketone (PEEK) lined Ti test cell die.20 The 77.5Li2S-22.5P2S5 glass SSE11,12 was used as the basis for all-solid-state construction was prepared by ball milling with an appropriate ratio of Li2S (Aldrich, 99.9%), reagent grade) and P2S5 (Aldrich, 99%) using a planetary ball mill (MTI Corporation SFM-1). The composite electrode was a 7:3 weight ratio of Sn powder (100 nm, Alfa Aesar, 99.9%) and glass SSE mixed by hand with agate mortar and pestle. 4 mg of the composite

*Electrochemical Society Active Member.

Present address: Battery Research & Development Division, LG Chem Research Park, Daejeon 305–738, Korea.

E-mail: kyuhwan@smu.ac.kr; sehee.lee@colorado.edu

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The Sn anode delivers a discharge capacity of 990 mAh g\(^{-1}\) C/100 is reached to allow for full lithiation. On the first cycle, the little Sn is electrically isolated. Additionally, pores are apparent in this leads to a continual interface between Sn active material and a dense, long-range network filling the porous SSE green compact. This leads to a continual interface between Sn active material and SSE where electrochemical reactions occur. It is evident that very little Sn is electrically isolated. Additionally, pores are apparent in the electrolyte layer, whereas the composite anode appears almost completely dense. In Figure 2b and 1c, EDS elemental maps and point spectra from Sn and SSE confirms the interpenetrating three-dimensional (3D) network structure. It has been well documented and highlighted in our previous studies that intimate solid-solid interfaces between active material and SSE are ideal for Li-ion and electron transport during discharge-charge processes.\(^{14}\)

Figure 2a presents performance of the composite anode over the first 50 cycles between 0.005 and 1.8 V at a C/10-rate (100 mA g\(^{-1}\)). Corresponding voltage profiles of the Sn:SSE electrode are shown in Figure 2b. A voltage hold was applied at 0.005 V until a current of C/100 is reached to allow for full lithiation. On the first cycle, the Sn anode delivers a discharge capacity of 990 mAh g\(^{-1}\) (respect to Sn) with a reversible charge capacity of 912 mAh g\(^{-1}\) exhibiting a high initial CE of 92%. This is an interesting result as charge recovery in Sn cells is usually very poor.\(^{24}\) A small irreversible capacity was identified at 1.1 V upon initial lithiation. This can be attributed to the decomposition of any native SnO\(_2\) layer as this is in line with previous reports and disappears on subsequent cycles.\(^{11}\) Subtracting out this contribution, the Sn alloys with 4.2 moles of lithium; this is close to theoretical capacity of Sn (959 mAh g\(^{-1}\)) and suggests the final crystallographic phase is the refined Li\(_{17}\)Sn\(_4\) phase.\(^{4,25,26}\) The exceptional initial CE also suggests that the side reactions are nonexistent. This is a different characteristic to conventional liquid electrolyte cells containing Sn which exhibit a continual formation of a solid-electrolyte-interphase (SEI) layer at the interface between active material and electrolyte.\(^{27}\)

For ease of reference, the voltage profile has been separated into distinct regions over the course of lithiation and delithiation and outlined in Table I. These correspond to the well-defined phase transformations of the Li-Sn alloy. The rest of this paper will use the region identifiers for simplicity. Due to operation at 60 °C, the identified potentials are actually closer to thermodynamic equilibrium than reports of other tin-based liquid cells.\(^{28}\) Surprisingly, the Sn composite displays exceptional specific capacity (>800 mAh g\(^{-1}\) Sn) and CE\(_{\text{Sn}}\) (99.2%) for the subsequent 50 cycles. From Figure 1a, we estimate an electrode thickness of 25 μm, which translates to a volumetric capacity of 844 Ah L\(^{-1}\).\(^{3}\) This is on the order of state-of-the-art silicon anodes in liquid cells.\(^{29}\) Outstanding capacity retention of the composite suggests the preservation of the networked microstructure, the presence of Sn and SSE.\(^{30}\)

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**Results and Discussion**

To determine the microstructure of the composite electrode, we used FIB to prepare a cross-sectional image of the uncycled Sn:SSE electrode (Figure 1a). Due to its inherent ductility, Sn powder forms a dense, long-range network filling the porous SSE green compact. To analyze the microstructure, cross-sectional samples were prepared from the as-pressed electrode before cycling test using a dual-beam focused ion beam (FIB, FEI, Nova Nanolab 200) equipped with energy dispersive X-ray spectroscopy (EDS).

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**Figure 1.** (a) FIB cross section view of pressed Sn:SSE composite anode on top of SSE separator layer. (b) EDS elemental maps indicating intertwining networked structure of Sn and SSE and (c) point EDS spectrum confirming presence of Sn and SSE.
lithiation hold is used which accounts for the reduced capacities). A general trend is seen that the initial lithiation and delithiation capacities decrease with decreasing pressure (Figure 3a). However, despite this characteristic, the first cycle CEs are essentially all the same for the three different pressures (Figure 3b). This would suggest that the pressures would simply be providing better active material utilization and not ensuring recovery of the original structure. This is further compounded by Figure 3d which demonstrates the first cycle voltage profiles at the three pressures. No discernable overpotentials are developed between the samples, however, lithiation Regions 3–5 tend to shorten. We suspect that with less confinement (ie. reduced pressure) more particle-particle connections are lost due to expansion of Sn. Thus there is a smaller amount of active material utilization at the highest lithiated phases of Sn with less pressure.

Another clear difference between the samples is the rate at which they achieve stability. With decreasing pressure it takes longer for the cell to achieve a stable CE, however, all samples reach close to 100% by the 10th cycle. A possible explanation for this can be determined through examining the resistance increase over the course of cycling as in Figure 3c. Resistance rise was calculated by taking the resistance difference between full delithiation and the uncycled electrode and multiplying by active material mass. This should negate any inherent contributions from the resistance of the electrolyte separator. The three pressures all achieve stability but resistant increments with decreasing pressure. This would indicate a loss of some electrical contact (Sn network) and ionic contact (SSE network) in the electrode thus supporting the claim of less active material utilization.

By the 10th cycle, the 0.05 MPa condition loses the characteristic Region 1 and 2 plateaus (Figure 3e). This is most likely attributed to the volume increase factors (VIF) presented in Table I. It can be seen that the cumulative VIF rise through Region 2 is 1.53. This would indicate that the Sn particles undergo a 53% increase in volume with the complete formation of LiSn. This is a rather miniscule amount compared to the ensuing expansion in Region 3. With the absence of an overpotential in Region 1 and 2 in the 10th cycle, this would indicate that some Sn becomes electrically isolated in the ambient pressure sample. Although, the Sn becomes reconnected once other particles expand enough to regain electrical contact. This behavior is interesting as it coincides with previous studies of monitoring in-situ pressure development over the course of Sn lithiation.30 Mukaibo et al. found no detectable tensile stress development until after the LiSn phase is formed and postulated a “stress accumulation” occurring in Region 1 and 2 leading to rapid volume change in Region 3. Once sufficient expansion has occurred in a number of the Sn particles, other Sn is reconnected and can undergo lithiation, rationalizing the very little change in the voltage profile of the ambient condition in Regions 3 to 5.

In analyzing the effect of pressure on lithiation processes of Sn in solid-state, we can see that this is mechanistically different than previous studies by Molina Piper et al. of applying higher pressures to simulate the confinement of silicon. The lower pressures used in this present study, allows us to study the preservation of electric and ionic pathways through particle contact rather than application of a strain energy.

In addition to examining the effect of pressure on formation cycles in the networked Sn electrode, it is also desirable to inspect the dual effect of pressure and rate on Sn kinetics. The networked Sn electrode was subjected to an asymmetric rate study with increasing delithiation rates and a constant C/20 lithiation rate. The delithiation voltage profiles were then split up into three distinct regimes: the first encompassing the reactions of Regions 4 and 5, the second covering Region 3, and the third comprising Regions 1 and 2. Figure 4a, 4b, 4c are the voltage profiles for the three different pressures at rates ranging from 50 mA g\(^{-1}\) (C/20) to 1000 mA g\(^{-1}\) (1C). Figure 4d plots the measured capacity contributions of the three delithiation regimes of the three pressures versus current density (insets highlight the regimes). It should be noted that there will be some additional error introduced in estimating capacity contributions at higher rates due to overpotential. However, designated Regimes can still be discerned since the voltage profile shape remains relatively intact.

**Regime 1:** Pressure and rate have the smallest impact on Regime 1. A constant difference in capacity of 20 mA h g\(^{-1}\) exists between the 20 MPa and 0.05 MPa samples for all currents. In all three samples, the capacity retention of Regime 1 is greater than 90% when normalizing to the C/20 rate. This can again be analyzed by looking at the VIF data in Figure 1. The cumulative change in VIF in Figure 1 is 0.74. Most likely the actual value is less than this amount as this assumes the full formation of Li\(_2\)Sn\(_3\). It has also been noted previously that electrochemical formation of Li\(_2\)Sn compounds in the range of 2.6 –

![Figure 2](https://example.com/figure2.png)

**Figure 2.** (a) Cycle performance and coulombic efficiency of Sn:SSE anode in all-solid-state Li-ion battery at 60°C with C/10-rate (100 mA g\(^{-1}\)) and (b) Corresponding voltage profiles of the Sn:SSE anode vs. LiIn counter electrode (converted to vs. Li). It is clear that formation of a stable structure occurs in the first 10 cycles.

**Table I. Summary of observed electrochemical lithiation regions of Sn in solid-state.** Crystallographic phases and volume increase factors are assigned from previous studies.\(^5\),\(^11\),\(^28\)

| Region | Potential vs. Li (V) | x in Li\(_x\)Sn | Crystallographic Phases | Volume Increase Factor |
|--------|---------------------|----------------|--------------------------|------------------------|
| 1      | 0.70 – 0.60         | 0 – 0.4        | Sn, Li\(_2\)Sn\(_3\)     | 1.23                   |
| 2      | 0.60 – 0.45         | 0.4 – 1.0      | Li\(_2\)Sn\(_5\), LiSn   | 1.53                   |
| 3      | 0.45 – 0.25         | 1.0 – 2.6      | LiSn, Li\(_2\)Sn\(_3\), Li\(_3\)Sn\(_2\), Li\(_3\)Sn\(_5\) | 2.81                   |
| 4      | 0.25 – 0.01         | 2.6 – 3.5      | Li\(_7\)Sn\(_6\), Li\(_7\)Sn\(_2\) | 2.98                   |
| 5      | 0.01 – 0.005        | 3.5 – 4.25     | Li\(_7\)Sn\(_2\), Li\(_7\)Sn\(_4\) | 3.55                   |

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Figure 3. (a) Specific capacity with respect to Sn for applied constant external pressures of 20 MPa (black), 10 MPa (blue), and 0.05 MPa (red), (b) Associated coulombic efficiencies with all reaching greater than 99.7% by the 10th cycle, (c) Change in delithiation resistance per cycle, normalized per gram of Sn, (d) 1st cycle voltage profile with dQ/dV spectrum, and (e) 10th cycle voltage profile with dQ/dV spectrum.

Figure 4. Asymmetric rate study of the Sn:SSE composite electrode with a constant lithiation rate of 50 mA g\(^{-1}\) and increasing delithiation rate up to 1000 mA g\(^{-1}\). (a) Delithiation voltage profiles for cell with external pressure of 20 MPa, (b) Delithiation voltage profiles for cell with external pressure of 10 MPa, (c) Delithiation voltage profiles for cell with external pressure of 0.05 MPa, (d) Capacity contributions of three distinct regimes of voltage profiles with increasing rate. Insets are provided to demonstrate regime inclusions.
x < 4.4 are highly disordered, this would lead to a smaller volume change than simulating the equilibrium phases. With a small volume change in Regime 1, this could ensure full utilization of active material and explain the high capacity retentions even with high rates. Stress development from the expanded Sn would simulate applying an external pressure which could explain the constant difference between the 20 MPa and 0.05 MPa samples. In another trend, the highest lithiated phases exhibit a decline in lithium diffusion coefficient and electrical conductivity, resulting in an increased overpotential with increased rate in Regime 1. Therefore, a disconnect exists between capacity retention and overpotential with rate.

Regime 2: Largely dominated by the transformation of Li2Sn to LiSn, Regime 2 exhibits the largest correlation amongst performance, pressure, and rate. It is clear that with decreasing pressure, the capacity drops significantly more with increasing rate. At 20 MPa, the cell retains 84% capacity at high rates whereas the 0.05 MPa cell retains 69% capacity. The VIF difference is 1.28, almost double Regime 1 and more than double Regime 3. Therefore, it is reasonable that the delithiation contribution declines with high rates. Dissimilarly, with the highest lithium mobility in this Regime, the overpotential associated with increased rate remains small. As Regime 2 demonstrates the greatest capacity loss yet smallest overpotential with rate, this further supports the claim of disconnect between the two.

Regime 3: This area exhibits very interesting characteristics, most notably that a limiting current is reached at about 200 mA g⁻¹, above which capacity contribution does not change significantly. This appears to be characteristic of Sn itself as all three pressures had very similar profiles. In analyzing the voltage profiles of Figure 4, all three pressures lost the distinct dual plateaus of Region 1 and 2 to blend together to form a single plateau most likely due to the lowest lithium ion mobility in this Regime. This Regime’s small VIF change (0.53) and previous studies showing small stress development coincide with our observation of sustained capacity even at high rates. It is interesting to note that the retention in this Regime is independent of the behavior in Regime 2.

From examining the codependence of pressure and current on delithiation mechanics, we can conclude that the most impacted factor is active material utilization. This is in direct correlation with volume increases in Sn as this serves to preserve electronic pathways. Therefore we see the highest impact of pressure and rate when Sn undergoes the largest volume changes. In the highest lithiated phases of the Li-Sn alloy, the large volume increases and stress development act to form an internal pressure within the electrode which negates the requirement for an external pressure. This is made possible by the inclusion of rigid solid electrolyte particles for Sn to expand into which separates the vast performance differences between solid and liquid cells containing Sn. Future work will focus on in-situ monitoring of Sn expansion in solid-state and try to determine the overall electrode level volume changes.

Conclusions

We have demonstrated the electrochemical performance of a high capacity Sn anode in an all-solid-state Li-ion cell. By taking advantage of the favorable inherent ductility and mixed conductivity of the active material, we formed an interpenetrating network of Sn and solid electrolyte. This ideally structured composite electrode delivers remarkable capacity of over 800 mAh g⁻¹ with respect to Sn and coulombic efficiency of 99.2%, while exhibiting a reversible Sn-Li alloying process. To our knowledge, this is the first report on utilizing high amounts of pure-Sn metal (70 wt% of the total electrode) in an all-solid-state Li-ion battery without any conductive additives; this allows for a volumetric capacity of 844 Ah L⁻¹ which competes with even some of the highest silicon-based systems. We investigated the effect of pressure and rate upon the delithiation mechanics of the favorable microstructure; correlations are drawn that volume increase factors and stress accumulation are the largest contributors to material utilization over the course of Sn-Li phase transformations. We even show that Sn develops a pseudo-pressure through expansion negate requirements for external pressure and allowing ambient operation – the next step toward commercialization of the solid-state battery. We believe that this initial work provides new opportunities to study the electrochemical expansion of Sn with the inclusion of rigid particles.

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Justin M. Whiteley, a Ji Woo Kim, a Chan Soon Kang, b Jong Soo Cho, a Kyu Hwan Oh, b and Se-Hee Lee a

aDepartment of Mechanical Engineering, University of Colorado at Boulder, Boulder, Colorado 80300-0427, USA
bDepartment of Materials Science and Engineering, Seoul National University, Seoul, 151-742, Korea

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