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Effect of Borophene on the Electrochemical Performances of $Li_7P_3S_{11}$ based All-Solid-State Lithium Sulfur Batteries

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Abstract

This study has investigated the effect of 2-dimensional (2D) beta borophene as a cathode additive for all-solid-state lithium-sulfur batteries. The comparisons have been carried out regarding the impact on ionic conductivity based on borophene content. Although the studies of borophene's contributions in the literature on the anode component, this study focuses on the cathode contribution for the first time. While MoS_2 has been selected as the cathode active material, carbon black has been selected as the electrical conductor, and $Li_7P_3S_{11}$ solid electrolyte has been synthesized as an ionic conductor in all-solid-state lithium-sulfur cells. Borophene has been synthesized from boron powder by the exfoliation method. As a cathode-active material, MoS_2, containing sulfur, and its 2D material nature, eliminates many of the disadvantages that sulfur exhibits when used alone. To investigate the effect of borophene on ionic conductivity in all-solid-state lithium-sulfur cells, multicomponent composite cathodes were prepared in (MoS_2 / Conductive Carbon / $Li_7P_3S_{11}$ + Borophene) overall compositions. According to the results, the specific capacity of the cells is affected negatively, while the stability of the cell is affected positively when increased the borophene amount.

Keywords: Energy storage, lithium sulfur battery, all-solid-state battery, borophene, $Li_7P_3S_{11}$ solid electrolyte

1. INTRODUCTION

The escalating demand for energy usage has had a direct impact on energy storage technologies. As a result, battery researchers are actively seeking novel materials. Notably, the recent exploration of 2D materials holds promise due to their enhanced diffusion rates, attributed to their distinctive two-dimensional surface structures. Graphene, one of the most widely recognized 2D materials, has gained popularity in next-generation batteries, owing to its exceptional columbic efficiency and augmented theoretical capacity (> 372 mAh g^{-1}). In the realm of lithium-sulfur batteries, 2D structures have emerged as essential considerations. Numerous studies have demonstrated the ability of these structures to effectively immobilize robust lithium polysulfide species. For instance, the 2D transition metal dichalcogenide ReS_2 has exhibited potential in sequestering lithium polysulfides, thus contributing to heightened cycling stability in Li-S cells [1].

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Solid-state lithium-sulfur batteries hold the potential to address many of the challenges posed by conventional Li-S batteries such as shuttle effect, polysulfide solubility, and safety concern [2]. Their enhanced safety, improved cycle life, and potential for higher energy density make them a compelling choice for the next generation of energy storage technologies. However, it's important to note that solid-state battery technology is still under development, and there are challenges related to manufacturing, cost, and performance that need to be addressed before widespread commercialization [3].

Borophene allotropes began to be estimated with the help of DFT simulations in the early 2010s, and towards 2015, they were synthesized on the Ag (111) surface [4]. Previously, 2D versions of boron were studied for other chemistry applications. These three allotropes were first synthesized as β12, χ3, and stripped borophene. All these allotropes have chemically active sites [5, 6].
Similar to other 2D materials, the synthesized borophene has proven to be remarkably intriguing and has emerged as a strong as a very good candidate for next-generation battery technologies due to its highly conductivity structures [7-9].

DFT simulations show that all three allotropes of borophene exhibit excellent anode properties, with theoretical capacities of 1984 mAh g^{-1} for β12, 1240 mAh g^{-1} for χ3, and 1239 mAh g^{-1} for stripped borophene [10, 11]. hold the potential for enhancing the performance of lithium-sulfur batteries. Among these allotropes, β12, with its superior theoretical capacity, stands out as one of the most frequently employed in batteries and serves as a focal point for more in-depth research [12]. Consequently, this allotrope is considered to be one of the most suitable candidates (attributed to its capacity and planar arrangement) for utilization as a cathode additive in solid cells [4, 12, 13].

This study revolves around the synthesis of $Li_7P_3S_{11}$ solid electrolytes by utilizing Li_2S and P_2S_5 components. These electrolytes have gained recognition as one of the most efficient solid electrolyte options for lithium-sulfur batteries within existing literature. However, challenges arising from interface issues with $Li_7P_3S_{11}$ electrolytes have led to performance setbacks in terms of ionic conductivity [14]. As a solution, researchers have turned their attention to exploring the impact of borophene additives (via partial replacement of the solid electrolyte) on the electrochemical cell's stability.

MoS_2, known for its unique and highly advantageous hollow sandwich structure, has gained widespread recognition for the significant positive impact it makes on enhancing both the ionic conductivity and electrochemical stability of lithium-sulfur cells [15]. This distinctive hollow sandwich structure of MoS_2 consists of a layered S-Mo-S sandwich structure reminiscent of graphite, held together by van der Waals forces with an interlayer distance of 0.62 nm [16-18]. This specialized configuration promotes the seamless accommodation and diffusion of lithium ions and polysulfide species throughout the battery's charge and discharge cycles, emphasizing MoS_2's potential as a versatile material for advanced energy storage applications.

Significantly, this structural framework accommodates a substantial number of Li-ions, thereby contributing to an impressive theoretical lithium storage capacity of 670 $mAh \cdot g^{-1}$ [16-19]. This property not only enhances the overall conductivity of the electrode but also minimizes the undesired trapping of polysulfides that often leads to performance degradation in traditional Li-S batteries. Given the exceptional characteristics offered by 2D MoS_2, it has been deliberately chosen as a cathode-active material for our study. By harnessing the inherent advantages of MoS_2's hollow sandwich structure, we aim to capitalize on its ability to mitigate common issues associated with lithium-sulfur battery systems.

This investigation, delving into the intricate interplay between the integration of borophene and the cathode performance of lithium-sulfur batteries, is poised to provide a fresh and comprehensive perspective to the scientific literature. This exploration not only leverages the unique attributes of MoS_2's structural design but also expands our understanding of how innovative materials like borophene can synergistically interact with MoS_2, potentially unlocking new avenues for further enhancing the efficiency and stability of advanced energy storage systems.

2. EXPERIMENTAL STUDIES

2.1. Material

Molybdenum disulfide (MoS_2), lithium foil, boron powders, and acetone were procured from Sigma Aldrich, Germany. For the synthesis of $Li_7P_3S_{11}$, intended for application in All-Solid-State lithium-sulfur cells, lithium sulfide (Li_2S, 99.98%, Sigma Aldrich, Germany) and phosphorus pentasulfide (P_2S_5, 99%, Merck, Germany) were employed as the initial constituents. These materials were meticulously stored within an argon atmosphere, maintained within a glovebox.

Other components required for the all-solid-state lithium-sulfur cell, essential for conducting electrochemical tests, were commercially sourced and utilized as-is.

2.2. Preparation of $Li_7P_3S_{11}$ Electrolyte

A mechanical activation method was employed to create the suitable $Li_7P_3S_{11}$ solid electrolyte. Initially, Li_2S and P_2S_5 (in a 70:30 molar ratio) powders were mixed in a planetary ball mill to attain an amorphous phase, subsequently undergoing crystallization at an optimized temperature. The chosen crystallization temperature directly impacts the ionic conductivity of solid electrolytes.

Optimal temperature values were determined in this study through heat treatments at varying crystallization temperatures. In the mechanical activation process, stoichiometric powder quantities were placed in a 20-ball (10 mm) zirconia pot and rotated intermittently at 400 rpm for 24 hours. The resulting ball-milled glassy powders were then sealed in a quartz tube and heated at 240-250-255-260-270 °C for 2 hours, with a heating rate of 10°C/min, to identify an appropriate crystallization temperature. The complete synthesis process was carried out within the glovebox to prevent powder exposure to air.

2.3. Synthesis of Borophene Powders

Borophene powders were synthesized to integrate the cathode into the lithium-sulfur cells. Initially, 50 mg of boron powders were homogeneously mixed for approximately 2 hours at room temperature using 50 ml of acetone and agate. Subsequently, the mixture was further stirred magnetically for an additional 2 hours. This dispersion was then transferred to an autoclave and maintained at 200 °C for 1 day. Following autoclave treatment, the dispersion was allowed to cool at room temperature for 6 hours, after which ultrasonication was performed with 225 W power. Subsequently, centrifugation was carried out at 8000 rpm for 15 minutes to separate the exfoliated particles.

2.4. Characterization of Prepared Materials

X-ray diffraction (XRD, Rigaku D-Max 200) was performed to carefully examine the crystallographic nature of synthesized $Li_7P_3S_{11}$ and borophene powders, using Cu K–alpha radiation in the region of 2theta of 10~90°. The structural morphologies of the borophene powders were investigated by field emission scanning electron microscopy (SEM, FEI FEG450).

2.5. Cell Assembling

The assembly of the cell proceeded as outlined below. To fabricate the electrolyte pellet, $Li_7P_3S_{11}$ powders underwent an initial cold-pressing step at a pressure of 360 MPa. The cathode material was formulated by amalgamating MoS_2, Conductive Carbon,

$Li_7P_3S_{11}$, and borophene. This composite cathode material was prepared with specific weight loadings of 30%, 30%, 20%, and 20%wt respectively. The synthesis involved consecutive ball milling phases, each spanning 10 hours. The objective of this approach was to bolster the ionic conductivity and foster improved interlayer contact between the cathode material and the solid electrolyte. For solid-state batteries, the incorporation of solid electrolytes onto the cathode side is a pivotal and customary procedure. Subsequently, Lithium foil was employed as the anode material, finalizing the cell construction.

2.6. Electrochemical Characterization

The electrochemical performance of the produced composites was be investigated by charge/discharge tests from in a custom-made electrochemical test cells with a 0.3 mA cm^{-2} current density in the 50–3000 mV (vs. Li/Li^+) range (MTI Model BST8-MA). The resistances of the cells were investigated using the ac impedance technique (EIS) with an amplitude of 5 mV and a frequency range of 1000 kHz to 0.1 Hz, (Gamry Instruments Reference 600).

3. RESULTS AND DISCUSSIONS

$Li_7P_3S_{11}$ can be prepared by many different methods using Li_2S-P_2S_5 [14]. It is known that this solid electrolyte has an ionic conductivity of over 10^{-4} S/cm at room temperature in lithium-ion batteries. In this structure, 70 or 75% Li_2S content is used in general, and crystallographically amorphous structures are obtained. These glassy powders are known for their superionic conductivity (1.7 $x10^{-2}$ S/cm) properties. The most important factor is whether these solid electrolytes are thermodynamically stable or not [20].
The XRD result of $Li_7P_3S_{11}$ glass ceramic synthesized by the mechanical activation method as a solid electrolyte for lithium-sulfur batteries is presented in Figure 1. The main characteristic diffraction peaks (2θ = 17.6°, 19.6°, 21.7°, 23.6°, 25.8°, and 29.6°) corresponding to the typical $Li_7P_3S_{11}$ phase, which is also in agreement with the literature and shows that the solid electrolyte $Li_7P_3S_{11}$ was produced successfully [21, 22].

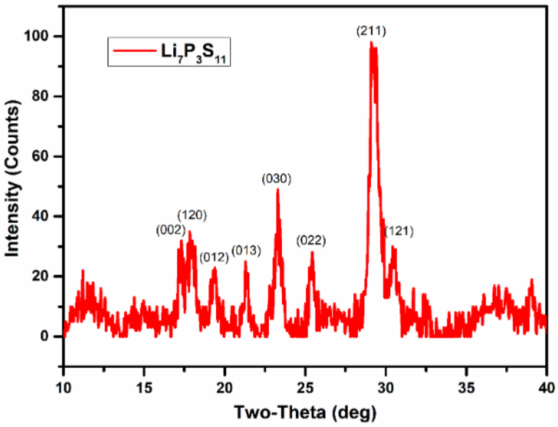

Figure 1 XRD analysis of $Li_7P_3S_{11}$ Glass Ceramic

The crystallographic structures of boron powders and borophene were characterized by XRD and compared in Figure 2. The characteristic peaks similar to those in boron powder are also observed in borophene. However, it can be said that the intensity of the peaks in borophene is slightly weaker than that of boron powders. This proves that the obtained structure is protected even after reduction. It is stated that the position of the diffraction angle between 25° and 30° of borophene is due to the distorted sp^2 hybrid orbitals of boron atoms [23].

Researchers have investigated the structure of β12 Borophene, and it has been determined that a=2.92621Å and b=5.06337Å in its unit cell. The structure is very unique and different even from other allotropes. The β12 borophene has a significant void concentration in its structure and seems to be the most suitable candidate for battery technology [24].

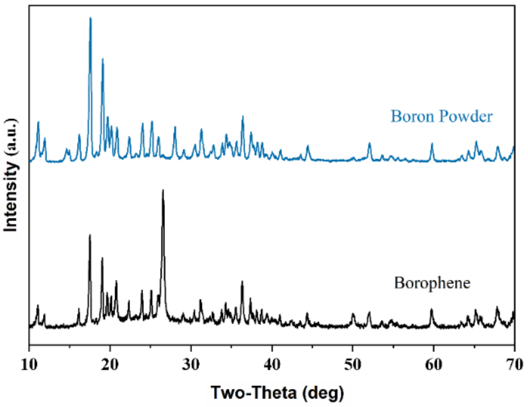

Figure 2 XRD analysis of Boron and borophene Powders

Figure 3 shows FESEM images of borophene powders. It was determined that borophene has a spherical fragmented structure. The average particle size of the borophene powders obtained is around 150 nanometers.

MoS_2 helps prevent the shuttle effect, which is one of the biggest problems of liquid-based lithium-sulfur batteries. It also enhances the ionic conductivity performance due to its two-layer structure. With 670 mAh g^{-1}, the theoretical specific capacity of MoS_2 is much higher than the active materials used in lithium-ion batteries. MoS_2 layers are formed by hexagonal covalent bonds (S – Mo – S). In other words, molybdenum atoms are located between the sandwich-like sulfur structure. This study performed XRD and SEM analyses to analyze the structural and morphological properties of MoS_2 powders for using lithium-sulfur cells.

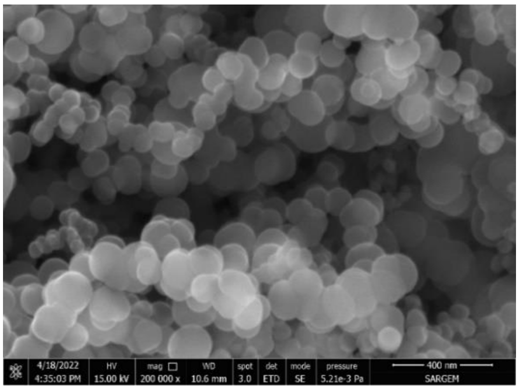

Figure 3 SEM analysis of borophene Powders

The crystal structure of MoS_2 was characterized by XRD analysis in Figure 4. The strongest peak is located at approximately 14° (002). In addition, the peaks at 33°, 39°, and 5°, respectively, corresponding to (100), (103), and (110) planes are observed for MoS_2 nanolayers. The absence of (002) reflection and the presence of narrow and sharp peaks are attributed to a single-layer or few multi-layer MoS_2 similar to other 2D materials [25, 26].

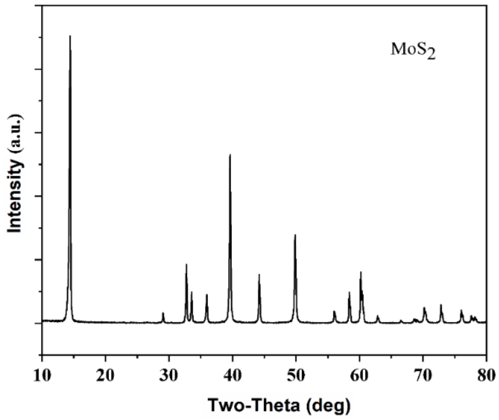

Figure 4 XRD analysis of 2D MoS_2 structure

The scanning electron microscopy (SEM) image presented in Figure 5 illustrates the fragmented and stratified structure of MoS_2 powders. This hierarchical arrangement is characterized by its dense and uniform appearance, albeit exhibiting agglomeration. The interlayer structure of MoS_2 holds significant implications for enhancing battery performance in liquid-based lithium-sulfur batteries. By impeding the diffusion of polysulfide species across its layers, the MoS_2 interlayer plays a pivotal role in ameliorating battery performance. When MoS_2 interacts with lithium, a transformative process occurs, leading to the formation of a distinct structure ($MoS_2 + 4Li^+ + 4e^- \rightarrow Mo + 2Li_2S$). Impressively, this configuration exhibits an energy density nearly twice that of graphene (372 mAh g^{-1}), underscoring its potential as a high-capacity energy storage material [27, 28].

MoS_2 films, characterized by high-density catalytically active edges, have emerged as a central focus of research in both liquid and solid-state sulfur batteries. Their ability to accommodate substantial sulfur content within their structure contributes to their prominence. In particular, the two-dimensional MoS_2 configuration offers an expansive surface area, notably within its thin layers, which facilitates the provision of additional active sites through the operation of weak van der Waals forces. This design confers stability to the cathode, effectively reducing interlayer resistance between the cathode and electrolyte. As a result, the kinetics of electrochemical reactions during cycling are enhanced, with implications for improved battery performance. The amalgamation of these attributes underscores the role of MoS_2 in the enhancement of lithium-sulfur battery technologies, heralding advancements in both energy storage and electrochemical performance.

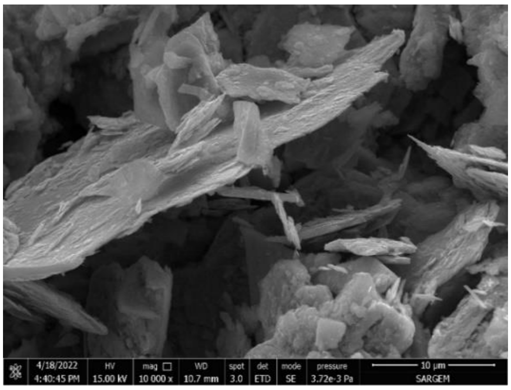

Figure 5 SEM analysis of 2D MoS_2 structure

In order to examine the effect of borophene on ionic conductivity in all-solid-state lithium-sulfur batteries, multicomponent composite cathodes were prepared in (MoS_2 / Conductive Carbon / $Li_7P_3S_{11}$ + Borophene) overall compositions. 30% MoS_2 active material and 30% carbon black conductive carbon amount of the cathode combinations were kept constant to investigate the effect of borophene on the stability of solid electrolytes. The remaining 40% is $Li_7P_3S_{11}$ + borophene mixture. Solid electrolytes and borophene were mixed in different amounts and added to the total cathode amounts. In this way, it was observed more clearly how borophene affects the ionic conductivity of lithium-sulfur all-solid-state batteries.

Figure 6 illustrates the Nyquist plots derived from the analysis of multicomponent composite cathodes. Within Nyquist measurements, the interplay of ion transfer mechanisms at the electrode/electrolyte interface during the Faradaic process is reflected, giving rise to distinct semicircular features evident in the impedance profiles. It is pertinent to note that the rapid pace of charge transfer processes can obscure the characteristic semicircular impedance pattern, leading to a dominant linear response. Additionally, the consistent and undisturbed nature of the electrode-electrolyte interface, lacking the irregularities necessary for semicircular impedance responses, further contributes to the prevalent linear behavior observed in the plots [29].

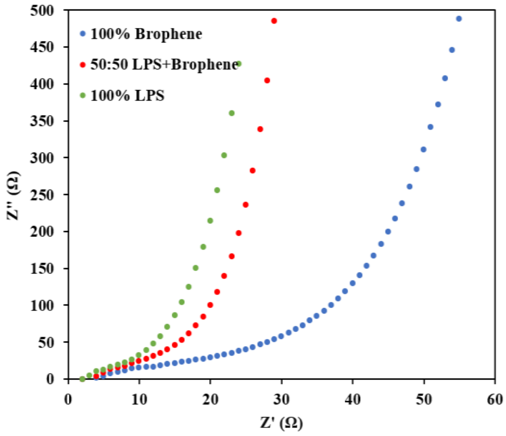

Figure 6 Nyquist plots of multicomponent composite cathodes

The Nyquist plot of the cathode composed entirely of $Li_7P_3S_{11}$ (100% LPS) reveals a distinctive linearity at low frequencies, indicative of robust charge transfer performance. In contrast, the cathodes with a composite composition of 50:50 LPS and borophene, as well as those constituted solely of borophene (100% borophene), exhibit diminished charge transfer performance. This disparity in performance can be attributed to the relatively lower ionic conductivity exhibited by borophene compared to $Li_7P_3S_{11}$, thereby influencing the charge transfer kinetics within the respective electrode-electrolyte systems [30].

According to the results, the specific capacity of the cells was affected negatively when the borophene amount increased. However, the stability and cycle life of the lithium-sulfur cells was improved. When $Li_7P_3S_{11}$ electrolyte is used 100% amount, the capacity of the cells increases up to the 10th cycle then a decrease is observed. This decrement continued gradually until the 50th cycle and completely faded its capacity at the end. The observed decrease in capacity can be ascribed to the heightened interfacial resistance evident between electrode and $Li_7P_3S_{11}$ solid electrolyte. Intriguingly, the derived diffusion coefficient governing this interaction was found to be orders of magnitude lower than the corresponding bulk diffusion coefficient for $Li_7P_3S_{11}$ [18].

When borophene is used in equal amounts with $Li_7P_3S_{11}$, a continuous increase in capacity is observed from the 1st to the 50th cycle. The increase in the specific capacity of the cells is directly related to the gradual activation. After the 50th cycle, the cell protected its stability until the 100th cycle but is not shown in Figure 7. This means that the activation process is completed and reflects the actual capacity of the cell after then.

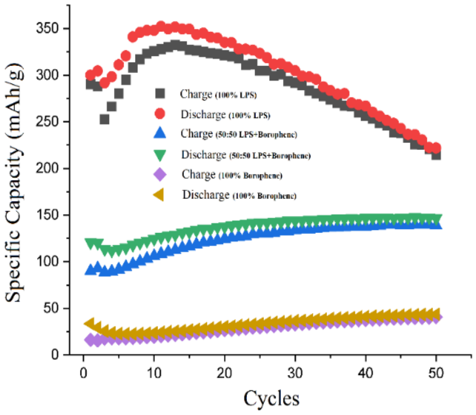

Figure 7 Electrochemical performance of lithium-sulfur cells

Electrochemical stability is also observed when borophene is completely substituted for the solid electrolyte. However, the capacity is observed as too low. It can be said that the contribution of full borophene to ionic conductivity is limited. According to the experimental results, equally using solid electrolytes and borophene contributes more positively to conserving capacity among all three combinations. But, in order to get more positive results, less amount of the borophene should be researched and reported for further studies.

4. CONCLUSIONS

Drawing from the comprehensive outcomes, it is evident that higher concentrations of borophene exert a discernible influence on the solid electrolyte, precipitating a significant decline in the cell's specific capacity. To counteract this undesirable capacity reduction and foster enhanced stability, a judicious strategy involves incorporating borophene into the solid electrolyte of lithium-sulfur cathodes at more conservative proportions. This approach aligns with the goal of preserving the cell's capacity while bolstering its overall resilience.

Moreover, the methodologies devised and executed within the purview of this study are characterized by their accessibility, practicality, and applicability. The straightforward implementation of these methodologies underscores their potential relevance in a broader context. In this regard, these findings hold promise in advancing novel paradigms for electrochemical energy storage, potentially serving as guiding principles for the trajectory toward the commercialization of all-solid-state lithium-sulfur batteries.

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The Declaration of Conflict of Interest/ Common Interest

The author has declared no conflict of interest or common interest.

Authors' Contribution

The author contributed equally to the study.

The Declaration of Ethics Committee Approval

This study does not require ethics committee permission or any special permission.

The Declaration of Research and Publication Ethics

The author of the paper declare that he comply with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that he does not make any falsification on the data collected. In addition, he declares that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic publication environment other than Sakarya University Journal of Science.

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