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The effect of the heat treatment to Co-N-C derived from ZIF-67 on the electrochemical performance of lithium-sulfur battery

Xiao Cui 🟫, Zheng-Fu Zhang 1,* , Jin-Song Wang 1 , Run-dong Wan 1 , Yu-Zhong Guo 1 , Juan-Ye Dao 1 and Chong-Jun Bao 1

1 Faculty of Materials Science and Engineering, Kunming University of Science and Technology, Kunming, 650000, People’s Republic of China
2 Kunming Metallurgical Research Institute, Kunming, 650000, People’s Republic of China
* Author to whom any correspondence should be addressed.

E-mail: zhang-zhengfu@163.com

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Abstract

The composite of porous carbon obtained from MOFs loading sulfur has been regard as promising candidate for lithium-sulfur batteries cathode material due to the large specific surface area, high porosity, diversity of types, and designable structures large specific surface area, high porosity, diversity of types, and designable structures. However, the production processes of MOFs materials play a pivotal role on the lithium-sulfur batteries performance. Herein, the effect of key process parameters such as heat treatment temperature, heat treatment time, and heat treatment atmosphere on the electrochemical performance of lithium-sulfur batteries were systematically studied. The results show that at a heating rate of 5 °C·min⁻¹, heat preservation at 900 °C for 3 h, and treatment with CO₂ airflow for 1 h, the composite material has the best electrochemical performance. The first discharge specific capacity of the lithium-sulfur battery reached 1060.32 mAh·g⁻¹, and after 50 cycles it reached 614.29 mAh·g⁻¹. This heat treatment research method based on MOFs-derived materials can be used as a reference for related research in other energy storage and conversion fields.

1. Introduction

The theoretical energy density (2600 W·h·kg⁻¹) and specific capacity (1675 mAh·g⁻¹) of lithium-sulfur battery (LSB) are significantly higher than that of the current commercial lithium-ion battery. And LSB becomes the most promising development direction of the next generation rechargeable energy storage system [1–3]. As cathode material, although elemental sulfur has many advantages such as availability, environmental friendliness and low cost [4, 5], there are a series of problems of LSB in practical application [6, 7]. First, the poor conductivity of sulfur and its final reduction products lead to inadequate initial discharge capacity; second, the volume change the conversion of Li₂S(discharge) and S(charge) would cause the volume change, thus destroying the balance of cathode structure and affecting the cycle performance and cycle life of battery; in addition, lithium polysulfides (Li₂Sₙ, 3 ≤ n ≤ 8) dissolve in liquid electrolyte and shuttle to cathode between the anode and the cathode, i.e., the so called 'shuttle effect' for lithium batteries, thus causing the consumption and corrosion of the anode [8–15]. Therefore, the cathode of LSB needs the composite material with a special structure design to make the LSB show better electrochemical performance [16, 17].

In recent years, organic-inorganic hybrid metal organic frameworks (MOFs) have attracted much attention and become one of the research hotspots and frontier directions in the field of new materials [18–21]. MOFs belong to a kind of crystalline structure materials, which can achieve a large specific surface area and obtain size controllable mesoporous and micropores [22, 23]. Open metal centers and heteroatom doping sites have strong adsorption capacity for lithium polysulfide (LIPS), as well as its advantages of simple synthesis and low cost, It has been studied as sulfur carrier materials [24–28].
Herein, the current study aims to improve the cycle performance and rate performance of LSB by using MOFs as sulfur carrier material at high temperature \cite{29, 30}. MOFs are prepared by liquid-phase reaction as sacrificial templates for cathode materials of lithium-sulfur batteries. A series of composite porous carbon materials with uniform cobalt and nitrogen doping are obtained by pyrolysis at high temperature under different heat treatment conditions \cite{30}. To obtain the best electrochemical performance of the composite porous carbon material heat treatment process, this study systematically investigates the effects of heat treatment temperature, time and atmosphere on the composition of electrode materials, the structure of composite materials, and the performance of lithium-sulfur batteries \cite{21, 31, 32}.

2. Experiment

2.1. Sample preparation

2.1.1. Synthesis of ZIF-67 precursor

A certain amount of cobalt nitrate hex hydrate and 2-methylimidazole were first dissolved in 100 ml methanol solution, respectively, ultrasonic for 30 min. After that, they were mixed and standing. Then the MOF material ZIF-67 was obtained by washing the powder sample with methanol solution for 3 to 5 times.

2.1.2. Preparation of Co-N-C porous carbon materials

ZIF-67 was filled in a crucible in an atmosphere furnace, then it was heated to 700 °C, 800 °C, 900 °C, 975 °C at a heating rate of 5 °C·min\(^{-1}\) in nitrogen atmosphere. After that, different MOF derivative materials were obtained after the temperature dropped down to room temperature.

2.1.3. Preparation of S/Co-N-C composite

The MOF was mixed with high purity sulfur powder in a mass ratio of 6:4, then evenly dispersed mixture powder was obtained by grinding. After that, the powder was placed in an inert gas protected sealed pot and kept at 155 °C for 18 h. Then the composite materials of S/Co-N-C composite were obtained after cooling.

2.2. Material characterizations

The microstructures of ZIF-67 and Co-N-C/S compound material were characterized by SEM analyses. The microstructures of the samples was characterized by Bruker D8 x-ray powder diffractometer. Focusing on the test conditions, the scanning range is 3\(^\circ\) < \(2\theta\) < 90\(^\circ\), and the scanning rate is 10\(^\circ\)·min\(^{-1}\). The specific surface area, pore volume and pore size distribution of the composites were measured by the specific surface area tester, and the pore types were analyzed by the adsorption desorption isotherms. The specific surface area tester used in the test is ASAP 2020, and the degassing pretreatment temperature is 300 °C.

2.3. Characterization of electrochemical performance

Xinwei BTS (5 V, 20 mA) battery test system was used for testing the constant current constant voltage charging and constant current discharge of the assembled batteries. The electrochemical performance was measured at a charge-discharge rate of 0.2 C (1 C = 1675 mA) and a test voltage of 1.7 V–3.0 V. The charge-discharge performance of the battery was tested with different rates. The cyclic voltammetry (CV) characteristics of LSB were measured by Shanghai Chenhua CHI760E electrochemical workstation, then the CV curve was obtained. The test voltage range is 1.7 V–3.0 V and the scanning rate is 1 mV s\(^{-1}\).

3. Results and discussion

Figure 1 (a) shows the synthesis of the S/Co-N-C composite. Firstly, the ZIF-67 precursor was used as the self-sacrificing template. The organic framework of ZIF-67 contains pyridine type nitrogen in 2-methylimidazole. The carbon materials with in situ nitrogen doping were obtained at a high temperature, and the pore structure was produced through pyrolysis. Porous carbon materials with Co particles surrounded by nitrogen-contained fragments were prepared by changing heat treatment temperature, holding time and inert gas constituent in the hearth. Then, elemental sulfur was injected into the pore structure of Co-N-C composite by melting method to form S/Co-N-C composite cathode material. Then the electrode sheet is fabricated and assembled into a battery, figure 1(b) shows the schematic diagram of LSB.

Figures 1(c)–(d) show the SEM images of the MOF material ZIF-67 which were enlarged to 50000 and 100000 times, respectively. The regular, ordered and smooth dodecahedron structure can be clearly seen in the figures, and the crystal particle size is about 5000 nm. It can be seen that the crystal surface is relatively flat and regular, the particle size is uniform, and there is sufficient surface area on the surface of the material as adsorption sites for active substances. Figure 1(e) shows the SEM image of ZIF-67 after heat treatment, which
has uniform particle size and good pore size distribution. Figures 1(f)–(g) presents the SEM images of Co-N-C/S composites, where sulfur elements can effectively penetrate into the porous material. It is obvious from the energy spectrum (figures 1(h)–(k)) that the Co, N, C and S elements are uniformly distributed.

Figure 2(a) shows the XRD patterns of the derivative materials obtained from ZIF-67 and the MOF material ZIF-67 after heat treatment at 700 °C, 975 °C for 3 h. In figure 2(b) (i.e., the X-ray diffraction pattern of ZIF-67 precursor), there is an obvious diffraction peak at 7.2°, indicating that the ZIF-67 crystal with good crystallinity is synthesized. After heat treatment, ZIF-67 crystals were annealed. Compared with the standard PDF card, the diffraction peaks of ZIF-67 are entirely replaced by the three characteristic peaks of Co. It can be seen from figure 2(c) that the adsorption isotherm of the Co-N-C sample belongs to type IV isotherm, with obvious hysteresis loops, indicating the existence of pore structure in the material. The adsorption capacity increases rapidly from the high-pressure area of the curve, and the curve is upwardly warped. This indicates that the accumulation mode of the material is irregular, and the material has a microporous structure. It is also consistent with the SEM image of the material, indicating that the specific surface area of the material has increased after heat treatment. The composite material has a good specific surface area (386.46 m² g⁻¹) and a rough surface. Figure 2(d) reveals that there are many microporous and mesoporous structures, which can be sulfur and polysulfides that can provide sufficient binding sites.

3.1. Effect of heat treatment temperature of ZIF-67 on electrochemical performance of LSB

The heating temperature is one of the important process parameters of the heat treatment process. The choice of the heating temperature is the main issue to ensure the quality of heat treatment. The holding time is 3 h, and the heating temperature is 700 °C, 800 °C, 900 °C and 975 °C, respectively. The first charge-discharge curve and the discharge specific capacity diagram of the first 50 cycles are shown in figures 3(a) and (b) below. It can be seen from figure 3(a) that different heat treatment temperatures have a certain influence on the first discharge specific capacity of the battery. The first discharge specific capacity at 700 °C, 800 °C, 900 °C and 975 °C is 827.91 mAh g⁻¹, 922.88 mAh g⁻¹, 1060.8 mAh g⁻¹ and 1201.01 mAh g⁻¹, respectively.

After 50 cycles (figure 3(b)), the specific discharge capacity is 506.94 mAh g⁻¹, 540.19 mAh g⁻¹, 614.29 mAh g⁻¹ and 510.99 mAh g⁻¹, respectively. The first discharge specific capacity increases with the increase of heating temperature from 700 °C to 975 °C. It is because the carbonization degree of MOF materials is gradually improved by higher temperature heat treatment. The electrical conductivity of the composite material after sulfur compounding enhanced the charge transfer at the interface. As a result, it improved the utilization efficiency of sulfur and the specific discharge capacity. The cyclic stability of the composites increased first and
then decreased, and reached the best value between 800 °C and 975 °C. The reason for this phenomenon is that the higher heat treatment temperature and the same heat treatment time make the material more carbonized, but it also destroys the original ordered structure of the material (figures S1(a)–(c) (available online at stacks.iop.org/MRX/8/055501/mmedia)), and the LIPS cannot be effectively adsorbed, which makes the specific capacity rapidly decay, thereby reducing the cycle stability of the material.

3.2. Effect of heat treatment time of ZIF-67 on electrochemical performance of LSB

Apart from the heating temperature, heat treatment time also affects the electrochemical performance of LSB. Reasonable control of the holding time can promote the completion of carbonization. The heating rate was 5 °C min⁻¹, the control heating temperature was 900 °C, and the holding time was 1 h, 3 h, 4 h and 5 h, respectively. The first charge–discharge curve and the discharge specific capacity of the first 50 cycles are shown in figures 3(c) and (d) below. As shown in figure 3(c), the first discharge specific capacity of 1 h, 3 h, 4 h and 5 h is 373.19 mAh g⁻¹, 922.87 mAh g⁻¹, 993.68 mAh g⁻¹ and 1047.28 mAh g⁻¹, respectively. It can be seen that the specific capacity of the sample holding for 1 h is significantly different from that of other samples. The charging curve shows an obvious overcharge phenomenon during the first charging process. The possible reason is that less heat treatment time makes the MOF under the same heat treatment temperature. The organic groups in the material are less carbonized, and the structure is relatively intact, which makes it inferior to other materials in conductivity, thus leading to the low utilization rate of active material sulfur in the cathode. Moreover, the short heat treatment time of the material makes the structure more complete, and there are no micropores or microcracks on the surface, which also leads to the load of the material itself. The ability of sulfur is reduced, and it leads to the agglomeration of sulfur, thus making the utilization rate of sulfur decrease in the initial discharge process.

After 50 cycles (figure 3(d)), the specific discharge capacity is 409.27 mAh g⁻¹, 500.17 mAh g⁻¹, 478.12 mAh g⁻¹, 413.04 mAh g⁻¹, at 1 h, 3 h, 4 h and 5 h, respectively. It can be seen that the cycling stability of the composites increases first, then decreases with the increase of heat treatment time and reaches the best value between 3 h and 4 h. The reason for this phenomenon may be that the short heat treatment time makes the MOF materials not enough pyrolysis degree, and the surface structure of the material itself is not suitable for dispersing sulfur (figures S1(d)–(f)). Its conductivity is also poor, which leads to the poor performance of the
material, and the long heat treatment time would cause excessive damage to the material. Although the conductivity is improved, the structure of excessive damage is not good. Besides, the dispersion of sulfur also has some effects.

3.3. Effect of ZIF-67 heat treatment atmosphere on electrochemical performance of LSB

By changing the inert gas to CO₂, not only can the active sites of the composite material be increased, but also it has the ability to effectively shorten the nitriding time. The heating temperature was 900 °C, and the holding time was 3 h. This study also investigated the effects of CO₂ treatment time on the properties of the products. The first charge-discharge curve and the discharge specific capacity diagram of the first 50 cycles are shown in figures 3(e) and (f) above. Figure 3(e) shows that the specific capacity of the first discharge increases slightly after applying a small amount of CO₂, but the specific capacity of the first discharge significantly decreases by applying CO₂ continuously. The possible reason for this phenomenon is that under the same heat treatment conditions, a small amount of CO₂ would oxidize the carbon structure on the surface of the material, resulting in micro defects [33]. This increases the specific surface area of the material, leading to the binding site of sulfur. As a result, this increases the utilization rate of the positive active substance and the specific capacity of the first discharge. However, when a large amount of CO₂ is oxidized at high temperature, it would lead to the formation of a large mesoporous structure on the surface of the material, and even break up the overall structure of the material. Finally, it would result in a rapid decline in electrical conductivity, thus affecting the electrochemical performance of the material and reducing the specific capacity of the first discharge.

After 50 cycles (figure 3(f)), the specific discharge capacity is 540.18 mAh g⁻¹, 606.77 mAh g⁻¹ and 413.04 mAh g⁻¹, at no inject CO₂, CO₂ 1 h and CO₂ 3 h, respectively. It can be seen from the curve trend that the cycling stability and discharge specific capacity of the sample without CO₂ is better than a large amount of CO₂, but worse than that of the sample with a small amount of CO₂. Overall, the effect of CO₂ on the electrochemical properties of the materials is not so large, and the performance of the samples would be slightly improved by adding a small amount of CO₂ for heat treatment.

After a series of research on heat treatment process, the heat treatment process was determined as follows: we heated something to 900 °C for 3 h at a heating rate of 5 °C·min⁻¹ under nitrogen condition, and then treated it with CO₂ for 1 h. After that, the MOF derivative Co-N-C composite was obtained, and then it was compounded...
with ordinary conductive carbon black in the ratio of 1:3 by thermal diffusion method to make LSB. The charge-discharge rate was 0.2 C, and the electrochemical performance was tested at a test voltage of 1.7 V - 3.0 V. The discharge specific capacity maps of the first 50 cycles are shown in figures 3(g) and (h). Figure 3(i) shows the first three CV curves of the LSB made of MOF derivative Co-N-C composite and sulfur under the optimized process conditions. The test condition of the CV curve is 1.7 V - 3 V and scanning rate is 1 mV s⁻¹. It can be seen from the CV curve that there are two similar oxidation peaks in the oxidation process of the first cycle, and the peak current of the oxidation peak first increases and then decreases during the charge-discharge process. According to the literature, it can be concluded that the reason is the bidirectional catalytic effect of nano Co particles in the process of charge-discharge reaction of LSB [34]. It can also be seen from the above figure that the position of reduction peak and peak current of Co-N-C composite in the first three cycles are not obvious, indicating that the discharge specific capacity of the material is relatively stable in the first three cycles. Moreover, it confirms that the carbon material containing element doping could enhance the cycle stability of LSB, which is consistent with the previous cycle stability curve.

4. Conclusion

In the current study, MOF ZIF-67 was used as a precursor to improve the conductivity of MOF by heat treatment, and sulfur composite was used as the cathode material of LSB. The effects of heat treatment temperature, heat treatment holding time and CO₂ gas flow rate on the morphology of MOFs, the structure changes of derivatives and electrochemical properties were studied by single variable control. The results show that at a heating rate of 5 °C min⁻¹, heat preservation at 900 °C for 3h, and treatment with CO₂ airflow for 1h, the composite material has the best electrochemical performance. The first discharge specific capacity of the LSB reached 1060.32 mAh·g⁻¹, and after 50 cycles, it has 614.29 mAh·g⁻¹.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

ORCID iDs

Xiao Cui https://orcid.org/0000-0001-9082-8976

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