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QILU UNIVERSITY OF TECHNOLOGY

本科毕业设计(论文)

Synthesis of MoTe_2 carbon cloth self-supporting flexible electrodes and performance study of lithium-sulphur batteries

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**Synthesis of MoTe₂ carbon cloth
self-supporting flexible electrodes and
performance study of lithium-sulphur batteries**

**MoTe₂ 碳布自支撑柔性电极
的合成及锂硫电池性能研究**

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ABSTRACT

Lithium-sulphur batteries, which has been studied by scientist for two decades, has high theoretical specific capacity of $1675\text{mAh}\cdot\text{g}^{-1}$, and high theoretical energy density of $2600\text{Wh}\cdot\text{kg}^{-1}$. However, the downside of lithium-sulphur batteries seriously limited the development of the lithium-sulphur batteries in last two decades. That is mainly because elemental sulfur and lithium sulfide has low conductivity, shuttle effect of lithium-sulphur batteries, and the sulfur anode volume change during the charging and discharging processes. Therefore, it is crucial to create a new cathode material that could release the problems above. Herein, in this paper we reported a MoTe_2/CC material by pretreatment of the carbon cloth and synthesis the MoTe_2 compound onto the carbon fiber. The SEM photo shows the MoTe_2/CC material was well synthesized, and the new Lithium-sulphur battery we prepared in the lab shows extreme high electrochemical properties from electrochemical workstation test. Therefore, the MoTe_2/CC material is a fantasist solution for the new lithium-sulphur batteries, and this material can be extended to apply for many different electrodes.

Key words: Lithium-sulfur battery; Characterization; Electrochemical performance, MoTe_2/CC ; Cyclic voltammetry

摘 要

锂硫电池经过科学家二十年的研究，理论比容量高达 $1675\text{mAh}\cdot\text{g}^{-1}$ ，理论能量密度高达 $2600\text{Wh}\cdot\text{kg}^{-1}$ 。然而，近二十年来，锂硫电池的缺点严重制约了锂硫电池的发展。这主要是由于单质硫和硫化锂的电导率低、锂硫电池的穿梭效应以及在充放电过程中硫阳极体积的变化所致。因此，创造一种新的阴极材料来解决上述问题是至关重要的。在此，我们通过预处理碳布，并在碳纤维上合成 MoTe_2 化合物，提供了一种 MoTe_2/CC 材料。SEM 照片表明， MoTe_2/CC 材料得到了很好的合成，电化学工作站测试表明，我们在实验室制备的新型锂硫电池具有极高的电化学性能。因此， MoTe_2/CC 材料是新型锂硫电池的理想解决方案，这种材料可以扩展适用于许多不同的电极。

Chapter 1. INTRODUCTION

1.1 Lithium-sulfur batteries background

At present, with the continuous depletion of the earth's energy and environmental pollution, as well as the development of wearable electronic devices, AI smart grid, intelligent textiles and artificial electronic skin, it is particularly important to develop a clean energy chemical energy storage system with high energy density.[1] Among various electrodes, flexible batteries, with excellent characteristics such as flexibility, stability and portability, have outstanding development potential in wearable electronic devices and small medical devices.[1] Recent studies on flexible batteries mainly focus on lithium-ion batteries with high energy density and cycle stability.[2] As a kind of lithium-ion battery, lithium-sulfur battery has sulfur as its cathode, and lithium metal as its anode, which completes the charging and discharging process through the mutual conversion of lithium elemental and lithium sulfide.[2] A high theoretical specific capacity of $1675\text{mAh}\cdot\text{g}^{-1}$ and a high theoretical energy density of $2600\text{Wh}\cdot\text{kg}^{-1}$ can be obtained for lithium-sulfur batteries. According to studies, the lithium-sulfur batteries also have the advantages of low cost, abundant sulfur reserves, and environmental friendly. However, the main reason that lithium-sulfur battery has not been applied in practice is that 1. Due to the existence of insulating elemental sulfur and lithium sulfide, the battery has poor conductivity, therefore, the actual discharge capacity of lithium-sulfur battery is low and the capacity attenuation is fast; 2. The polysulfide generated by charging and discharging reaction is easily dissolved in liquid electrolyte, resulting in shuttle effect, which led to reduction of utilization of active sulfur, and the generation of lithium dendrites; 3. In the process of charge and discharge, the sulfur anode produces nearly 80% volume change, which will cause the electrical contact between the electrode material and the collector to deteriorate and increase the internal impedance.[3][4]The factors above will lead to the problems of the lithium-sulfur batteries in short cycle life, fast capacity decay and low coulomb efficiency of lithium-sulfur batteries. The self-supported sulfur electrode has good ion and electron conductive skeleton and more negative sulfur ion carrier sites, so the self-supported material as the sulfur element carrier of lithium sulfur battery can effectively increase the surface load and content of sulfur in lithium sulfur battery.[5] Therefore, the positive sulfur electrode prepared by the self-supporting flexible material is expected to improve the electrochemical activity, reduce

the polarization, relieve the volume expansion in charge and discharge, enhance the adsorption of polythium sulfide to reduce its shuttle effect, and thus significantly increase the actual capacity, rate and cycle stability of the lithium-sulfur battery. Carbon cloth has high electrical conductivity and good mechanical strength, and is a good substitute for traditional aluminum foil, which can be directly used in the fluid collection of self-supporting cathode materials for lithium-sulfur batteries [6]. Based on the pore structure formed by interweaving carbon cloth, the carbon cloth impregnated with active sulfur and then heat-treated at 155°C has good chemical properties, high cycle, high specific electric capacity and so on. These advantages make carbon cloth a promising electrode material.[2]

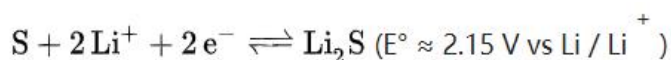
Carbon cloth has the characteristics of high conductivity and good mechanical strength, so it can be used to replace the traditional aluminum foil as a collecting fluid in the self-supporting sulfur anode material of lithium sulfur batteries.[1] From this perspective, more and more carbon cloth self-supporting electrodes are used in lithium-sulfur batteries based. Recent research related to self support lithium-sulfur batteries based on carbon cloth material is multiple. Elazari Prepared ACF-S anode material by soaking the carbon cloth into active sulfur. The material has advantages of high sulfur surface load, good electrochemical performance, and high specific discharge capacity; Miao prepared CFC/S anode material by carbonizing the carbon cloth with nitrogen environment. Which has 96% of capacity retention ratio. Lei grew the WS₂ nanosheet on carbon cloth by hydrothermal method to get the self-supporting collection fluid (C@WS₂). This material could efficiently adsorb poly-sulfide, and result in the improval of electronic conductivity.

However, the non-polar surface of carbon-based materials has a very limited adsorption effect on polar lithium polysulfide, which makes the intermediate lithium polysulfide hinder the conductivity of the battery, resulting in the decrease of the final positive electrode sheet conductivity and lower battery performance than estimated.[8] Therefore, it is one of the major challenges in the field of lithium-sulfur batteries to design and prepare anode materials that can effectively inhibit the shuttle effect.

1.2 Cyclic voltammetry

Cyclic voltammetry (CV) is a potentiodynamic measurement in electrochemistry.

Which is generally used to test the electrochemical properties of molecule that is adsorbed onto the electrode[11]. While the cyclic voltammetry is operating at a electrochemical workstation. During the experiment, the current at the working electrode is plotted versus the applied voltage. In the cyclic voltammetry method experiment, the electric potential increases as the time goes. Which causes the redox reaction in both cathode and anode of the battery. The current in cathode decreases as the concentration of reducible analyte reduced, and the reduced analyte would begin to be reoxidized during the reverse current. The redox reaction of lithium-sulfur battery shows in equation 1. During this process, the electric potential between the working electrode and the reference electrode, and the current between the working electrode and the reverse electrode are measured by the electrochemical workstation. These data can be used to draw the current-potential figure. On the cyclic voltammetry figure, the more similar the shape of the oxidation peak to the reduction peak means the higher reversibility of the charging and recharging process. Therefore, the cyclic voltammetry figure could show the information of redox potential and electrochemical reaction rate.[10,11]



Equation 1. Redox reaction of lithium sulfur battery

1.3 Battery cycle life and its affection

Battery cycle refers to the number of charge and discharge cycles that a battery can complete before losing proper performance. The battery cycle life is affected by discharge rate, temperature, and cut-off voltage. However, due to internal oxidation reaction, which can cause the internal resistance increase. Therefore, the battery could not release the stored energy, the battery life gradually ends. There are several reasons that could shorten the lithium-sulfur battery cycle life. 1. Lithium metal deposition. For some reason, partial of the lithium ion could not form the stable compound during the charging process. Instead, lithium ion obtain one electron and become lithium metal. This phenomenon could reduce the electric capacity of the battery. 2. Shuttle Effect. In the charging and discharging processes, polysulfide compound appear as by product on the anode of the battery. These polysulfide could move freely to the cathode, and react with lithium metal. Which could seriously reduce the conductivity of battery, and cause short battery life. 3. Electrolyte depletion. In the process of continuous circulation, due to the limitation of chemical and thermal stability

electrolyte decomposition and volatilization will occur. Which cause the electrolyte could not be fully infiltrated into anode and cathode material, reduce the battery cycle life. [12]

1.4 Electrochemical impedance spectroscopy (EIS)

Lithium sulfur battery has higher potential on anode and lower potential on cathode compared with other batteries. During the charging and discharging processes, the lithium ion remove from cathode and embed on the cathode. As a result, the charge-discharge capacity, cycle stability and charge-discharge ratio of anode and cathode materials for lithium-ion batteries are closely related to the process of lithium ion removal and embedding in chimeric electrode materials. Electrochemical impedance spectroscopy could easily show the process of removal and embedding of lithium ion. An EIS is consisted of 4 parts. 1. high frequency area: the semi-circle related to the diffusion and migration of lithium ions through SEI film on the surface of active material particles. 2. medium and high frequency area: A semicircle relating to electron transport within a particle of an active materia. 3. Medium frequency area: The semicircle associated with a charge transfer process. And 4. low frequency area: An line related to the solid diffusion process of lithium ions within the active material particles. By analyzing the intercept point of EIS spectroscopy we can conclude that the lower medium intercept shows the better cyclic stability and electrochemical performance.[13][14]

1.5 MoTe₂ material

Molybdenum ditelluride (MoTe₂) is compound of molybdenum and tellurium. The MoTe₂ could crystallize in two dimensional sheets, which can be thinned down to flexible and transparent monolayers. MoTe₂ materila is a semiconductor, and its band gap is in the infrared region, which increases the possibility of using as a semiconductor in electronics or an infrared detector. N-type MoTe₂ material has a electrical conductivity of $8.3 \Omega^{-1}\text{cm}^{-1}$ with 5×10^{17} mobile electrons per cubic centimeter. P-type MoTe₂ shows conductivity of $0.2 \Omega^{-1} \text{cm}^{-1}$ and hole concentration of $3.2 \times 10^{16} \text{cm}^{-3}$. Powder MoTe₂ has more resistance. In the high current situation, MoTe₂ material shows negative resistance. As the current increases, the voltage at both ends of the material decreases. Which means as the increase of current, the potential between the material could decrease.[17] As a result, attaching the

MoTe₂ material to the cathode of the lithium sulfur battery could increase the conductivity of the battery, rise the energy capacity and increase the battery stability and cycle life. These features of MoTe₂ material could also release the lithium sulfur battery problems on cathode, reduce the impact of shuttle effect on the battery.[18]

Chapter 2. METHOD

2.1 Preparation of lithium-sulfur battery electrode

2.1.1 Hydrophilic treatment of carbon cloth

The carbon cloth (1x1 cm²) was cleaned three times with deionized water, acetone and isopropanol respectively via ultrasonic treatment. Then the carbon cloth was put into a flask, and soaked in a homogeneous 1:1 volume mixture of concentrated sulfuric acid and concentrated nitric acid for 2 hours. Followed by refluxing the flask at 80 °C in water bath for 1 hour. After that, the carbon cloth was rinsed with deionised water and ethanol till pH is 7, and dried at room temperature.

2.1.2 Synthesis of MoTe₂/CC material

0.346g Tetrahydrate and ammonia molybdate ((NH₄)₂Mo₇O₂₄·4H₂O) and 26ml N,N-dimethylformamide (O=C(N(CH₃)₂)₂) were added into reaction kettle, then the carbon cloth was added. The reaction kettle was heated at 200°C in a hydrothermal oven for 12 hours. Element Molybdenum (Mo) was connected to the carbon cloth after this reaction. The carbon cloth was cooled down until room temperature. Ethanol was used to rinse the carbon cloth to get rid of the impurities on the surface of carbon cloth. Until no red impurities drop off from the carbon cloth, it is dried at room temperature. The carbon cloth was carried by a porcelain boat, and put into a tube furnace in air conditions. Meanwhile, the carbon cloth was heated at a rate of 5°C per minute, until the temperature rises to 400°C, keep the temperature for 2 hours. Molybdenum oxide was formed on the carbon cloth during this process. The carbon cloth was cooled down at room temperature. 0.2g tellurium powder (Te) was add on the both sides of carbon cloth. The carbon cloth was put back to the tube furnace on a porcelain boat. The tube furnace was vacuumized by argon and hydrogen for 3 times to remove the air. After which, the carbon cloth was heated in the argon and hydrogen condition at 620°C for 3 hours. MoTe₂ was formed on the carbon cloth during this reaction.

1 mg sublimed sulfur and carbon cloth was put into the reaction kettle. The carbon cloth was rinsed by carbon disulfide solution (CS₂) to dissolve the solid sulfur. Stand the reaction kettle for 5 minutes. After that, the disulfide solution was volatilized in a hydrothermal oven. The reaction kettle was put into a container, and heated at 155°C for 12 hours. The MoTe₂/CC material was obtained. The MoTe₂/CC material that obtained was scanned by scanning electron microscopy to observe the characterization of the material.

2.2 SEM

The scanning electron microscope (SEM) is a scanning microscope that could produce the images by scanning the sample by using the electrons high energy beam.[20] When the electrons interact with the scanning sample, characteristic X-rays was produced as a signal. Then the signal is collected by the detectors and form the images. The resolution of SEM depends on many factors. For instance, the electron spot size and interaction volume of the electron beam with the sample. Some of SEM could provide less than 1 nm resolution. However, typical modern SEM could provide the resolution of 1-20 nm. The SEM is one of the most widely used methods used for the examination and analysis of the micro and nanoparticles. [15] The characterization analysis of MoTe₂/CC was observed by scanning electron microscope.

2.3 XRD

X-ray diffraction analysis (XRD) is a X-ray technology widely using in material science. By irradiating the sample material with incident X-ray, use the detector to receive and measure the scattering angles and intensities of the X-rays that transmits the material. The spectrum that shows on screen could determine the crystallographic structure of a material. X-ray diffraction analysis method could determine multiple of the material structure properties, that includes lattice parameters, strain, grain size, epitaxy. Phase composition, and preferred orientation. In addition, the XRD method could also be used to identify crystalline phases and orientation, determine atomic arrangement, and measure thickness of thin films and multi-layers.[16] On the spectrum of XRD, the broad humped peak position refers to the amorphous material with short range ordering. Instead, a sharp peak on the XRD

spectrum means the material is crystalline. By compare the XRD spectrum with typical orthorhombic phase of the material, the synthetic material could be analyzed. [19]

2.4 Assembly of lithium sulfur battery and its electrochemical performance test

Positive electrode shell, MoTe₂/CC material, 20ul of electrolyte, membrane, 20ul of electrolyte, lithium tablet, and cathode shell were assembled separately from top to bottom of the lithium-sulfur battery in the glove compartment in argon and hydrogen condition. The battery was pressed by 500N once after its finishing assemble. After which, a voltammeter was used to test the electric tension of the lithium-sulfur battery. 2 to 3 volt means the battery is proper functioning. LAND battery testing system was used to do the constant current charge-discharge test of the battery in room temperature. Scanning speed of 0.1 mVs⁻¹ to 0.5 mVs⁻¹ of cyclic voltammetry curve were tested separately by electrochemical workstation. The impedance spectrum of the lithium-sulfur battery was tested as well.

Chapter 3. RESULT

3.1 Characterization analysis of MoTe₂/CC

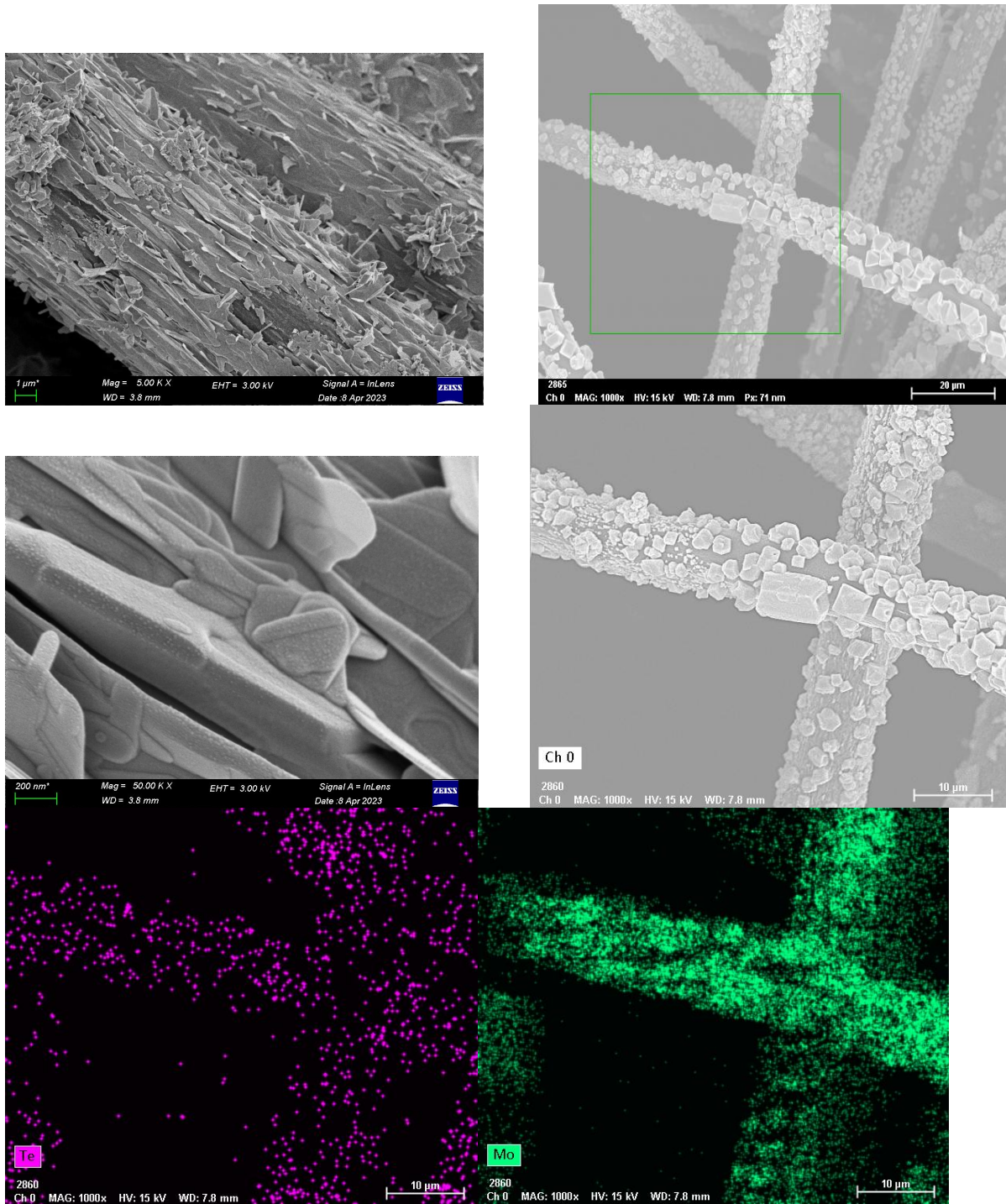


FIGURE 1. (a,b) High magnification SEM images of MoTe₂ coated on carbon cloth

fiber. (c) SEM image and photograph of carbon cloth material fiber. (d-f) HAADF-STEM image of single MoTe₂/CC fiber and Ch O, Te, and Mo elemental mapping.

As displayed in the high-magnification SEM images in Figure 1a, 1b, many of MoTe₂ two-dimensional sheet like aggregation nanocones self assembled on the carbon cloth fiber. Figure 1c show the SEM image of carbon cloth fiber fully covered with the MoTe₂ compound. Figure 1(d-f) show the HAADF-STEM image and Ch O, Te, and Mo elemental mapping results in each MoTe₂/CC nanocone. As a result, the MoTe₂ compound were formed onto the carbon cloth fiber properly. The structure and morphology of MoTe₂/CC material is favorable for electron transfer.

In addition, the structure features of MoTe₂/CC can be further demonstrated by a representative X-ray diffraction (XRD) pattern. The XRD pattern that determined shows in figure 2. The MoTe₂/CC characteristic diffraction peaks can be proper match to the typical orthorhombic phase of MoTe₂ compound. Which is well match to the SEM images.

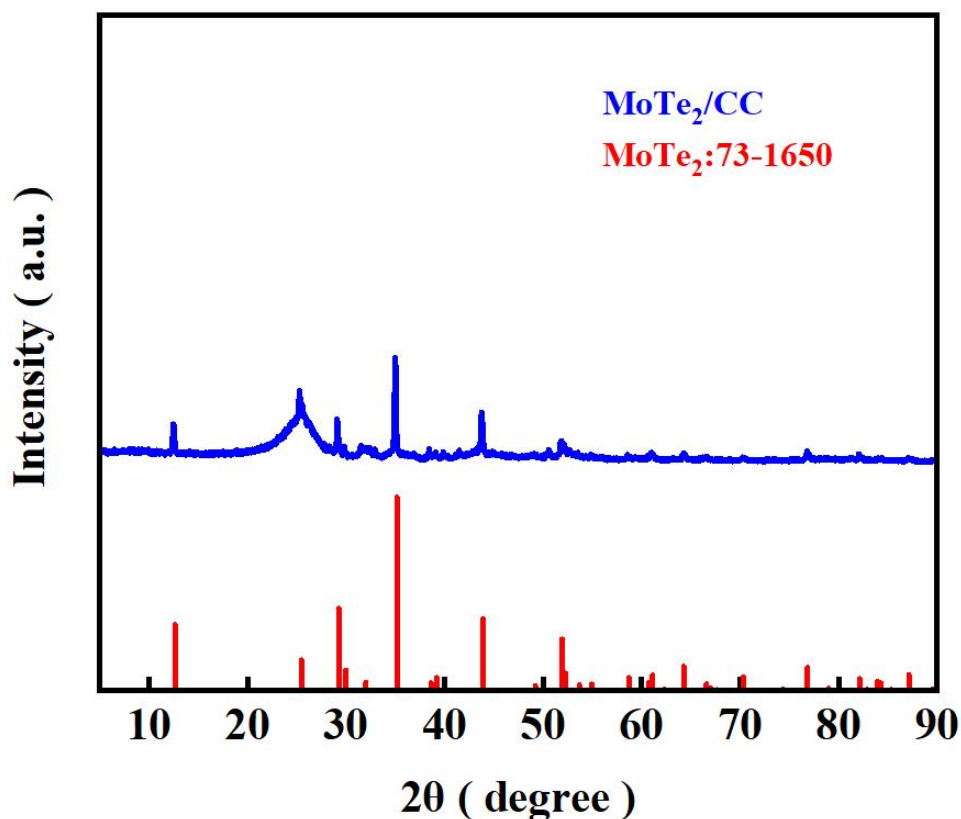


FIGURE 2. Representative X-ray diffraction (XRD) pattern of MoTe₂/CC material.

3.2 Electrochemical performance of MoTe₂/CC

According to figure 3, the electrochemical impedance spectroscopy (EIS) plots of MoTe₂/CC lithium sulfur battery and blank sample display an inclined line at low frequency (less than 1Hz). That is because diffusion of the solid state protons in the protons electrode. In the medium frequency and high frequency, the EIS curves consist of a semicircle, which means the charge transfer resistance. The intercept at higher frequency means the ohmic resistance. Based on the fitting of equivalent, the battery uses MoTe₂/CC material has less charge transfer resistance than the blank CC lithium sulfur battery, which decreases from 76.91 ohm to 58.58 ohm. Due to the good electrical conductivity properties of MoTe₂, the lithium-sulfur cells can get fast electron conduction and fast ion transport properties. As a result, using MoTe₂/CC material can enhance the rate capacity and recycle stability.

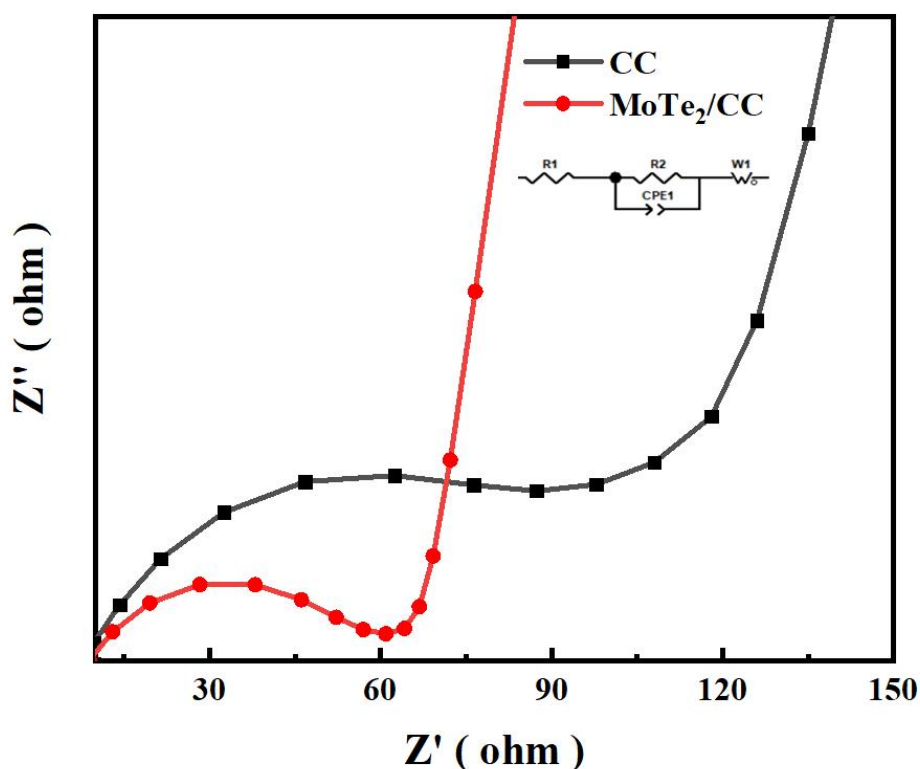


FIGURE 3. The (EIS) plots of MoTe₂/CC lithium sulfur battery and blank sample (CC/S).

Figure 4 and 5 shows the cyclic voltammogram of MoTe₂/CC battery and CC/S battery at a scanning rate of 0.1-0.5 mVs⁻¹. It is easy to conclude that the both of the samples have stronger peaks on cyclic voltammogram when the scanning rate is higher. The peak of MoTe₂/CC battery that shows on the cyclic voltammogram is stronger than the peaks shows on blank sample CC/S battery. Which indelicate that the MoTe₂/CC battery has fast kinetics for lithium sulfide conversion in the cathode. Compared with CC/S battery, the anodic peak of MoTe₂/CC battery has a trend of moving to lower potential in general. Instead, the cathode peak of MoTe₂/CC battery move to the higher potential compared with CC/S battery. This phonomonal shows that the MoTe₂/CC battery support efficient lithium sulfide redox reactions and faster kinetics throughout cycling.

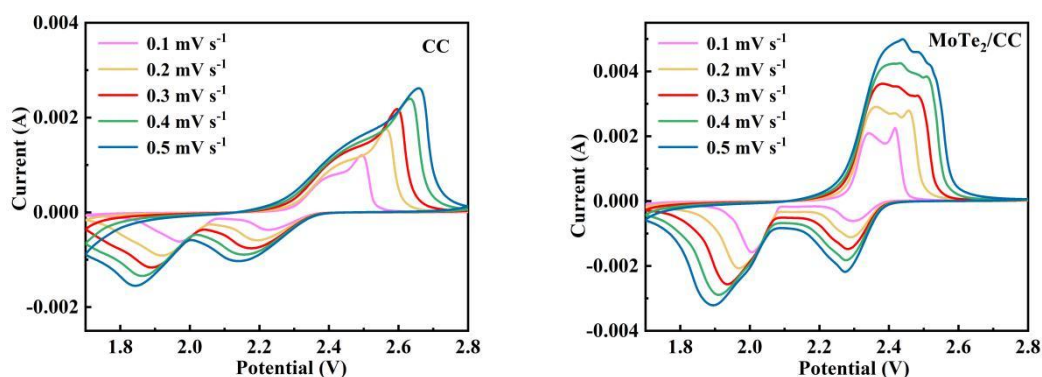


FIGURE 4,5. Cyclic voltammetry curve of MoTe₂/CC lithium sulfur battery and CC/S lithium sulfur battery as blank sample.

MoTe₂/CC lithium sulfur battery shows the favourable cycling performance at 1C in figure 6. In which the initial discharge specific capacity of 917 mAhg⁻¹. The discharge specific capacity decreases to 645 mAhg⁻¹ after 100 cycles. The coulombic efficiency has a high level of over 99.27 percent. Due to the the activation of sulfur material of MoTe₂, the specific capacity is gradually increase. Compared with the blank carbon cloth sample shows in figure 7, the MoTe₂/CC lithium sulfur battery shows more specific capacity in general. As a result, MoTe₂ compound attached on the carbon cloth could significantly increase the specific capacity of the battery.

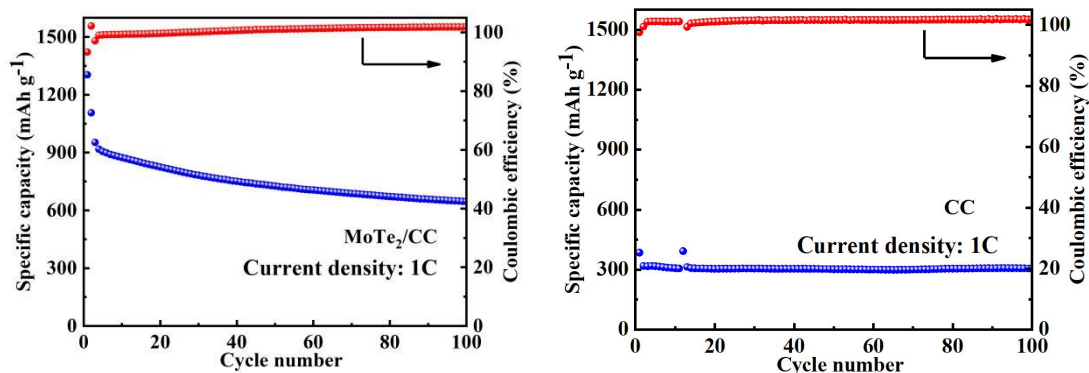


FIGURE 6,7. Battery cycle of MoTe₂/CC lithium sulfur battery and the CC lithium sulfur battery as blank sample.

The constant current charge and discharge curve of lithium sulfur battery samples shows in figure 8. The charge and discharge plateau of the MoTe₂/CC lithium sulfur battery at 0.2C is considerably long compared with the blank carbon cloth sample. The MoTe₂/CC lithium sulfur battery shows the greater capacity of 1304 mAhg⁻¹ compared with blank sample at 424 mAhg⁻¹. Besides, the battery has MoTe₂ shows lower oxidation potential plateaus, and smaller potential gap between the oxidation and reduction plateaus. Furthermore, the battery with MoTe₂ shows lower interlayer than the blank sample battery (ΔE). Which means when the conductive interlayer used in lithium sulfur battery, the transfer between electrodes becomes effective. Besides, in figure 8, the lithium sulfide nucleation point of MoTe₂/CC battery shows higher than CC/S battery (2.2V). Which indicates MoTe₂/CC battery is more stable, and can store more electric energy.

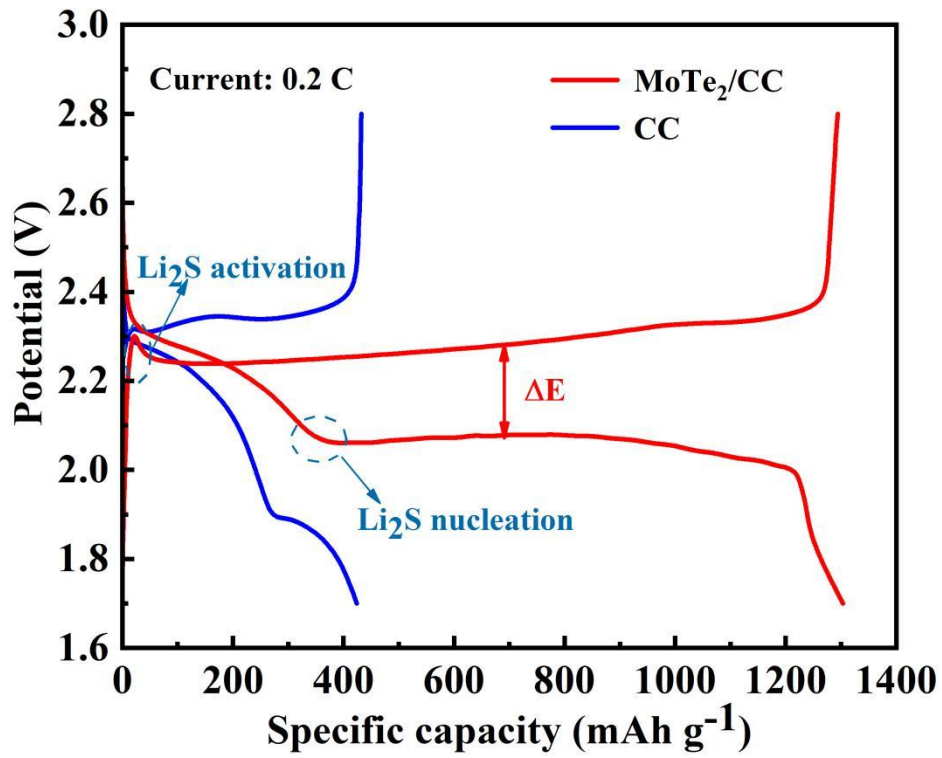


FIGURE 8. Constant charge and discharge of MoTe₂/CC lithium sulfur battery and its blank sample in 0.2C.

Chapter 4. CONCLUSION AND DISCUSSION

In this experiment, MoTe₂ was successfully attached on to the carbon cloth fiber after pretreatment and synthesis of MoTe₂/CC material. The characteristic SEM image and X-ray diffraction (XRD) pattern can prove the successful of the syntheses of the MoTe₂ material, and the material was well grew on the carbon cloth fiber. And the MoTe₂/CC material is favorable for electron transfer and has good electrical conductivity properties. Besides, the electrochemical test was successfully done with the electrochemical workstation. The result of electrochemical impedance spectroscopy (EIS), battery cycles, clavanostatic charge and discharge, and cyclic voltammetry curve were determined. The MoTe₂/CC battery has less charge transfer resistance, high capacity and recycle stability, longer battery cycle life, have faster kinetics of cycling and effective transfer between electrodes.

However, the MoTe₂/CC material was prepared in the lab, and only characterization and electrochemical experiment was done. None of the practical testing was finished, the MoTe₂/CC material is in a theoretical position. Besides, more parallel experiments could be done in the further researches. Therefore, more practical and electrochemical experiment could be done in the future.

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