Multifunctional Co$_3$S$_4$/Graphene Composites for Lithium Ion Batteries and Oxygen Reduction Reaction

Nasir Mahmood, Chenzhen Zhang, Jie Jiang, Fei Liu, and Yanglong Hou$^{*[a]}$

Abstract: Cobalt sulfide is a good candidate for both lithium ion batteries (LIBs) and cathodic oxygen reduction reaction (ORR), but low conductivity, poor cyclability, capacity fading, and structural changes hinder its applications. The incorporation of graphene into Co$_3$S$_4$ makes it a promising electrode by providing better electrochemical coupling, enhanced conductivity, fast mobility of ions and electrons, and a stabilized structure due to its elastic nature. With the objective of achieving high-performance composites, herein we report a facile hydrothermal process for growing Co$_3$S$_4$ nanotubes (NTs) on graphene (G) sheets. Electrochemical impedance spectroscopy (EIS) verified that graphene dramatically increases the conductivity of the composites to almost twice that of pristine Co$_3$S$_4$. Electrochemical measurements indicated that the as-synthesized Co$_3$S$_4$/G composites exhibit good cyclic stability and a high discharge capacity of 720 mA h$^{-1}$ up to 100 cycles with 99.9% coulombic efficiency. Furthermore, the composites react with dissolved oxygen in the ORR by four- and two-electron mechanisms in both acidic and basic media with an onset potential close to that of commercial Pt/C. The stability of the composites is much higher than that of Pt/C, and exhibit high methanol tolerance. Thus, these properties endorse Co$_3$S$_4$/G composites as auspicious candidates for both LIBs and ORR.

Keywords: cobalt · electrochemistry · lithium ion batteries · nanotubes · oxygen reduction reaction

Introduction

Electric vehicles (EVs) and hybrid electric vehicles (HEVs) are emerging as a new type of transport to combat the high cost of oil and environmental challenges. High-performance energy storage devices with both high energy and high power densities are prerequisites for such vehicles. Of the devices currently available commercially, lithium ion batteries (LIBs) attract tremendous attention because of the state-of-the-art technology incorporated into these high-energy batteries. LIBs have been used for various portable electronic devices, such as laptops, personal digital assistants, and cellular phones. However, it is still a great challenge to achieve high-power densities in LIBs for EVs and HEVs. In general, the power capability of LIBs is hindered by kinetic problems in the electrode materials as they are not capable of releasing all their energy in a few seconds. For high rate and power densities, the electrode materials in LIBs must possess high rates of ionic and electron diffusion, which can be achieved by shortening the diffusion path or by the use of buffering materials. New electrode materials are being investigated extensively to make a combination of anode and cathode that can achieve the goals proposed by the U.S. Advanced Battery Council (USABC).

Fuel cells are another type of storage device, for example, proton exchange membrane fuel cells (PEMFC) and direct methanol fuel cells (DMFC), which have great potential as clean and efficient power sources for both EVs and portable electronics due to their high energy conversion efficiency, low operation temperature, low or even zero emission, and high energy and power density. But the major challenge associated with fuel cells is the high cost of the catalyst required for the cathodic oxygen reduction reaction (ORR). To reduce the cost of fuel cells, the replacement of platinum with nonprecious metals or metal-free catalysts is necessary and some effort has been made to prepare platinum-free catalysts with promising performance, for example, carbonized catalysts, cobalt- or iron-based catalysts, tungsten carbides, and transition-metal chalcogenides. Transition-metal chalcogenides have been used in many fields, for example, in optical sensors, optoelectronic devices, magnetic devices, and as catalysts. Cobalt sulfides with different stoichiometric ratios, such as Co$_{1-x}$S, CoS$_2$, Co$_3$S$_4$, and Co$_9$S$_8$, are the most important type of metal chalcogenides. These cobalt sulfides have shown great potential for use in LIBs, semiconductors, magnets, catalysts, and photocatalytic degradation. The thermal stability and electrical conductivity of cobalt sulfides are much better than those of other metal sulfides, however, they have a number of major drawbacks, namely...
capacity fading, low conductivity, and poor cyclability; these are associated with the pulverization of electrodes, which results in the decay of the specific capacity and the formation of polysulfide anions. These polysulfide anions dissolve in the organic solvent of the electrolyte, which causes the low conductivity. Also, the polysulfide anions migrate across the separator membranes to the cathode side leading to the poor cyclability. To resolve these problems, we have employed graphene as a conducting matrix as well as a buffering substrate. Apart from increasing the electrical conductivity, graphene with a large surface area and high flexibility also improves the contact between the active material and the electrolyte, and protects the active material from structural changes. The amorphous nature of graphene is helpful for absorbing the polysulfide anions, which prevents them from dissolution in the electrolyte. Poizot et al. described the mechanism for the reaction of sulfides with Li⁺ as shown in Equation (1).

$$M^{n+} + ne^- + nLi^+ \rightleftharpoons M^0 + nLiX, X = S, O$$

(1)

In this work, composites of Co₃S₄ nanotubes (NTs) and graphene (Co₃S₄/G) have been synthesized and their applications in LIBs and ORRs explored. To the best of our knowledge, composites of graphene and Co₃S₄ NTs applied to LIBs and ORR have not yet been reported. It was found that the introduction of graphene by a simple hydrothermal method into the Co₃S₄ system enhances both thermal stability and electrical conductivity of Co₃S₄. The novelty of this work is the investigation of the multifunctionality of the Co₃S₄/G composites and the resolution of the problems associated with chalcogenides stated above. Multifunctional composites of Co₃S₄ NTs and graphene have been synthesized by a modified literature method by incorporating graphene into the cobalt substrate as illustrated in Scheme 1. The multifunctionality of the composites results from the peculiar tubular morphology of Co₃S₄ and the incorporation of graphene, which leads to strong electrochemical coupling and an increased electrical conductivity in the composites. The composites show a better performance than all other types of cobalt sulfides, and better than other chalcogenides used in LIBs.

**Results and Discussion**

The sacrificial $\text{Co}^\text{(CO)}_{0.35}\text{Cl}_{0.20}\text{(OH)}_{1.10}$, Co₃S₄, and Co₃S₄/G composites were structurally characterized by powder X-ray diffraction (PXRD) analysis. The XRD patterns show that all the products are crystalline, no impurity or other phases are observed (Figure 1a). All the peaks are well indexed to the standard card of the sacrificial $\text{Co}^\text{(CO)}_{0.35}\text{Cl}_{0.20}\text{(OH)}_{1.10}$ (JCPDS No. 38-0547) and to the pure cubic phase of Co₃S₄ in both the pristine form and the composites (JCPDS No. 47-1738). The broad peaks observed for Co₃S₄ and the composites reflect their very fine grain size. Very weak peaks are observed in the case of the composites at 2θ = 16 (111) and 2θ = 220 (20); graphene also shows broad peaks at the same points, which indicates its amorphous nature. It can be anticipated that the presence of graphene affects the intensities of these peaks. In the case of Co₃S₄, one peak at around 2θ = 35° is observed, which can be indexed to another phase of cobalt sulfide (CoS₂ or Co₄S₃), which indicates the pure phase Co₃S₄ of the products.

Raman studies were carried out to confirm the presence of Co₃S₄ NTs on the graphene sheets. Figure S1 in the Supporting Information clearly shows that as graphite oxide (GO) is converted into graphene, the intensity of the D band increases. The D band shows an even higher intensity due to the presence of NTs on the graphene sheets, which causes more disordering in the graphene planes. X-ray photoelectron spectroscopy (XPS) was employed to study the synthesis of the composites; the spectra are shown in Figure S2. The XPS studies confirmed the presence of C, O, Co, and S elements in the composites with no other element incorporated. The elemental analysis of graphene is presented in Figure S3a and reveals the presence of oxygen in the graphene. To check which type of oxygen is present, further XPS characterization was performed. Figure S3b shows different types of functionality are present with the oxygen and carbon atoms in the forms C–C (284.80), C–O (286.10), C=O (287.78), and COOH (288.89 eV). Thus, the presence of these functionalities contributes to the better performance of the Co₃S₄/G composites as elaborated in the respective results.

Thermogravimetric analysis (TGA) was performed in air in the temperature range of 25 to 800°C to measure the Co₃S₄ and graphene contents in the composites (see Figure S4). Note that the weight loss at around 80°C corresponds to the presence of moisture in the sample. A major weight change occurs in the range of 440 to 600°C as a result of removal of graphene by oxidation. Finally, the small weight increase observed is a result of the oxidation of cobalt because the TGA studies were performed in air. The TGA data indicates that the Co₃S₄/G composites contain 21% graphene and 79% Co₃S₄.
The transmission electron microscopy (TEM) image of Co(CO)\textsubscript{0.35}Cl\textsubscript{0.20}(OH)\textsubscript{1.10} shows wire-like nanostructures (Figure 1b), which serve as the sacrificial templates for the growth of Co\textsubscript{3}S\textsubscript{4} NTs. It is confirmed that the product mainly contains crystallized wires of Co(CO)\textsubscript{0.35}Cl\textsubscript{0.20}(OH)\textsubscript{1.10} with an average diameter of 300 nm and several microns in length. The high-resolution TEM image also reveals that the Co(CO)\textsubscript{0.35}Cl\textsubscript{0.20}(OH)\textsubscript{1.10} wires grow in such a way that the layers stack compactly upon one another, which might produce wires with a high aspect ratio, as shown in the inset of Figure 1b. Figure 1c shows a representative image of the as-synthesized Co\textsubscript{3}S\textsubscript{4} NTs with an average diameter of 300 nm and lengths of a few microns. The high-resolution TEM image in Figure S5a in the Supporting Information illustrates that these NTs are produced by very fine tiny particles arranged in a tubular fashion. The PXRD peaks also demonstrate the polycrystalline nature of the NTs. The growth of these NTs is based on the Kirkendall effect, in which cobalt diffuses to the surface where it reacts with sulfur from thioacetamide (TAA) as the sulfur source.\textsuperscript{[37]} The NTs are porous, which might be favorable for the diffusion of Li\textsuperscript{+} into the NTs. The better performance of these NTs in LIBs and the ORR may partially be attributed to this porosity (see below). It is well known that the porosity may arise from different solid-state diffusion rates of the reactants.\textsuperscript{[37]} It has been suggested that the Co\textsubscript{3}S\textsubscript{4} NTs form as a result of the faster diffusion of the cobalt ions outwards rather than inwards, which leads to vacancies in the sacrificial templates that connect to produce the voids that result in the tubular structures. The morphology of the composites is shown in Figure 1d, from which it is clear that the NTs have good adhesion and dispersion in the graphene matrix. Although some particles remain on the NTs and destroy their tubular morphology as depicted in the TEM images in Figures 1d and S5b, the NTs do not breakdown significantly and thus would not affect the performance of the composites. The high-resolution TEM (HRTEM) image (Figure 1e), a magnification of the area enclosed by the dashed lines in the inset,
shows the crystalline planes of Co₃S₄. The lattice spacing \(d\) of 0.236 nm can be assigned to the (400) plane of Co₃S₄. The selected-area electron diffraction (SAED) pattern (Figures 1f and S3c), which indicates that the Co₃S₄ NTs that grow on the graphene sheets are crystalline, and the diffraction rings can be assigned to the corresponding crystalline planes of Co₃S₄. Thus, the TEM analysis further confirms the morphological structure of the Co₃S₄/G composites, that is, Co₃S₄ anchored on to the graphene sheets (gray), as shown in Figure 1d,e and the inset of Figure 1e.

The electrochemical performance of the as-synthesized Co₃S₄/G composites as an anode material for LIBs was studied by using the galvanostatic discharge/charge technique. Figure 2a shows the 1st, 2nd, 10th, and 20th cycle discharge/charge curves of the composites at a current density of 0.2C (1C = 702.8 mAh g⁻¹) over the voltage range 0–3 V versus Li⁺/Li. The initial discharge and charge capacities were 2151 and 1449 mAh g⁻¹, respectively. The reason for the large initial discharge and charge is the formation of the solid electrolyte interface (SEI) at the surface of the electrode as a result of the decomposition of the electrolyte and the incorporation of graphene, which improves the electrode/electrolyte contact.[10] The capacity of the composites decreased in the first four cycles and then became stable at around 720 mAh g⁻¹ up to 100 cycles with a coulombic efficiency of around 99.9%, as shown in the Figure 2b. This obvious capacity fading in the initial cycles is a result of many complicated side-reactions and the irreversible insertion of Li⁺ into the composites as well as the formation of the SEI.[23] In comparison, Co₃S₄ showed discharge and charge capacities of 1134 and 770 mAh g⁻¹, respectively (Figure S6a in the Supporting Information). Continuous fading in the capacity was observed with increasing number of cycles, as shown in Figure 2b. Note that the composites showed better performance at the higher rates of 0.5C and 1C than the pristine Co₃S₄ at 0.2C (Figure 2c). The performance of the composites at 1C was stable and showed almost twice the capacity of Co₃S₄ at 0.2C and maintained a capacity of up to around 350 mAh g⁻¹ even after 100 cycles. The much higher stability and performance of the composites can be attributed to the presence of graphene, which offers a number of benefits, such as acting as an elastic buffer and thereby accommodating volume expansion and shrinkage changes during the uptake and release of Li⁺ and thus preventing structure cracking and crumbling of the electrode.[43] Also, graphene provides an electronically conductive substrate,
better electrochemical coupling between the composites and electrolyte, and a surge in the conductivity of the electrode, which result in higher cycling performance, rate capability, and capacity.

Cyclic voltammograms for the Co$_3$S$_4$/G composites are presented in Figure 2d and show the oxidation and reduction changes that occur during the insertion and extraction of Li$^+$. The cyclic voltammetry (CV) measurements were carried out at a scan rate of 0.2 mV s$^{-1}$ in the potential range of 0–3 V versus Li$^+$/Li. In the first cycle, two reduction peaks are observed at 0.6 and 1.0 V, and two oxidation peaks at 2.0 and 2.5 V. In addition to these two oxidation peaks, another small peak is observed at 1.2 V. These reversible peaks strongly affect the capacity and cyclic stability of the electrode. In subsequent cycles, the main cathodic peaks at 0.6 and 1.0 V shift towards the higher potentials of 1.1 and 1.6 V, and similarly, the anodic peaks shift slightly negatively. The decrease in the next cycle is consistent with the charge and discharge curves, reflecting the capacity fading in subsequent cycles. The shift of the main cathodic peak towards a higher potential in the following cycles corresponds to a multistep electrochemical reaction of lithium with the electrode that leads to the formation of a SEI film on the surface of the electrode, decomposition of the electrolyte, and the formation of Li$_2$S.[44,47,48] It also explains the first high discharge of the composites. No peak was observed in the cyclic voltamgram at 1.2 V for Co$_3$S$_4$ (see Figure S6b in the Supporting Information); this oxidation peak appears only in the voltammograms of the composite electrode as a result of the insertion/extraction of lithium into the graphene. Therefore it is confirmed that the graphene present in the composites is also electrochemically active towards lithium.[43] It has previously been reported that the charge/discharge process is an important factor affecting the cyclic performance of cobalt sulfide.[46] The charge/discharge curves of the composites (Figure 2a) show that two significant chemical changes occur during the intercalation of Li$^+$ into the product, which can be accounted for by reactions (2) and (3), and are supported by the CV studies, which show that some decomposition occurs at 1.6 V.

\begin{align}
\text{Co}_3\text{S}_4 + \text{Li}^+ + \text{xe}^- & \rightarrow \text{Li}_x\text{Co}_3\text{S}_4 \\
\text{Co}_3\text{S}_4 + 8\text{Li}^+ + 8\text{xe}^- & \rightarrow 4\text{Li}_2\text{S} + 3\text{Co}
\end{align}

To investigate the reason why the Co$_3$S$_4$/G composites exhibit an electrochemical performance that is so much higher than the pristine Co$_3$S$_4$ electrode; the influencing effect of the graphene has already been investigated in several systems.[44,47,48] Graphene also reduces the charge transfer resistance, which has been attributed to the active sites on the electrode material. The graphene has some carboxy groups on its surface (as confirmed by XPS), which facilitate the production of more active sites on the surface of the electrode and lowers the charge transfer resistance.[43] To determine the actual values, the Nyquist plots were further investigated as both curves have two semicircles at high/medium frequency and a straight line at low frequency. The semicircle in the high-frequency region corresponds to the SEI film resistance ($R_1$), the semicircle in the medium-frequency region corresponds to the charge transfer impedance ($R_2$), and the inclined line is related to the diffusion of Li$^+$ (Warburg impedance, $W$).[49] By fitting the equivalent circuit, as shown in the inset of Figure 3, the SEI film resistance and charge transfer resistance were calculated. Values for the SEI film resistance and the charge transfer resistance of 16.64 and 45.20 $\Omega$ cm$^2$ for the pristine Co$_3$S$_4$ electrode and 7.58 and 25.66 $\Omega$ cm$^2$ for the Co$_3$S$_4$/G composite electrode, respectively, were determined. These values confirm that graphene enhances the cyclic performance and capacity of the composites. In the equivalent circuit, $R_3$ is the ohmic resistance of the electrolyte and cell components, and the CPEs are the phase element constants that should be considered in the nonhomogeneous condition of the composites.

The ORR catalytic activity of the Co$_3$S$_4$/G composites was analyzed by charging the composites on to the glassy carbon (GC) electrode for CV measurements in both 0.1 m KOH and 0.5 m H$_2$SO$_4$ (see Figure S7 in the Supporting Information). CV was performed both in an oxygen- and nitrogen-saturated environment to investigate the ORR performance of the composites. The CV behavior, that is, the much lower current density with no peak observed under nitrogen as compared with oxygen, confirms the pronounced ORR activity of the composites in both basic and acidic media.

![Nyquist plots for the Co$_3$S$_4$ and Co$_3$S$_4$/G composite electrodes in the range of 100 kHz to 10 MHz.](image3)
Figure 4. a) ORR polarization curves for the Co3S4/G composite and Pt/C electrodes in 0.1 \text{m} KOH solution. b) ORR polarization curves for the Co3S4/G composites before and after acid treatment in 0.5 \text{m} H2SO4 solution.

Figure 4a shows the ORR polarization curves for the composites and commercial Pt/C in 0.1 \text{m} KOH. The onset potential for Co3S4/G at around $-0.05 \text{ V}$ is close to that of Pt/C ($-0.02 \text{ V}$) at 1600 rpm, which suggests that the composites could be a promising candidate for the cathodic ORR. The polarization curve for the composites in 0.5 \text{m} H2SO4 at 1600 rpm is shown in Figure 4b. The composites in 0.5 \text{m} H2SO4 exhibit a broad region associated with a mixed kinetic-diffusion mechanism and ill-defined diffusion-limiting current, so that Pt/C may have little advantage over the composites. The smaller surface area of the composites might lower the performance, whereas the commercial Pt/C has a large surface area. Note that the performance of the composites could be improved by leaching the composites with 0.5 \text{m} H2SO4 at 60\text{°C} for 8 h prior to the test for ORR in acidic media. As shown in Figure 4a, it is generally accepted that the increase in the performance after leaching is due to the removal of H2SO4 of the ORR inactive species present in the composites.\(^{[24]}\) In 0.1 \text{m} KOH, the current density reaches $-4.5 \text{ mA cm}^{-2}$ close to $-1.1 \text{ V}$ versus Ag/AgCl, which reveals the four-electron transfer mechanism. To investigate the kinetics of the oxygen reduction, polarization curves were obtained at different rotation speeds (see Figure S8a,c in the Supporting Information). An increase in the rotation speed leads to an increase in the diffusion of oxygen at the surface of the electrode, which results in large current densities. The four-electron mechanism for the reduction of O2 to H2O is very important for the application of fuel cells, and so to determine the number of electrons, Koutecky–Levich (K–L) plots were used (see Figure S8b,d) for both basic and acidic media. The more negative the potential is, the larger the electron transfer number ($n$), approaching four. In 0.1 \text{m} KOH, the electron transfer number at $-0.5 \text{ V}$ versus Ag/AgCl is 3.2 and at $-1.1 \text{ V}$ versus Ag/AgCl is 3.9, whereas in 0.5 \text{m} H2SO4, the electron transfer number at 0.20 \text{ V} versus Ag/AgCl is 2.0 and at $-0.10 \text{ V}$ versus Ag/AgCl is 3.2. These values of $n$ indicate a mixture of two- and four-electron mechanisms for ORR at the composite electrodes in both basic and acidic media.

The durability of the composites was tested in 0.5 \text{m} H2SO4 over 20000 s and it was observed that the composites are much more durable in acidic medium than commercial Pt/C (see Figure S9 in the Supporting Information). The relative current of the composites, which was measured by constant current galvanostatic discharge after 20000 s, is above 60\%, but for Pt/C it is only 10\%. Such high durability of the composites is due to the presence of graphene, which provides a better conducting substrate and good electrochemical coupling between the substrate and oxygen molecules, and thus facilitates the ORR through the facile transport of electrons between the oxygen molecules and the active sites of electrode.

One of the major drawbacks of commercial Pt/C in the cathodic ORR is that its performance is significantly affected by the addition of methanol.\(^{[31,50]}\) Thus, methanol tolerance was investigated for the Co3S4/G composites under the same conditions as Pt/C in both acidic and basic media (Figure 5). To test the tolerance of the composites, 2\% (w/w) of methanol was used and it was found that methanol has no effect on the performance of the Co3S4/G composites at the cathode. Although the onset potential of the Co3S4/G composites is slightly lower, its high durability and remarkable methanol tolerance give it superiority over commercial Pt/C and thus it is a better candidate for the cathode ORR.

**Conclusion**

A simple template-based hydrothermal method has been used to develop Co3S4/G composites, which exhibit good dispersion, alignment, and adhesion of Co3S4 NTs in the graphene matrix. The higher performance of the composites is based on the porous structure of Co3S4 and the conductive and elastic nature of the graphene matrix, which enable both Li$^+$ and e$^-$ to migrate and occupy all the active sites of the composites and protect it from structural changes during the insertion and extraction of Li$^+$. All these attributes make it an attractive candidate for use as an electrode material both in LIBs (with an initial discharge of 2151 mAh g$^{-1}$) and for cathodic ORR. The beauty of this work is that a
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**Experimental Section**

**Synthesis of graphene:** Graphene was prepared from graphite oxide (GO). In a typical synthesis, GO (80 mg) was sonicated in water (30 mL) for 30 min and then NaOH (8 g) was added and the mixture shaken well to homogenize the mixture. This mixture was then transferred to a 50 mL Teflon-lined stainless-steel autoclave and the reaction was allowed to proceed to homogenize the mixture. This mixture was then transferred to a 50 mL Teflon-lined stainless-steel autoclave and the reaction was allowed to proceed in the oven for 12 h at 200°C. After completion of the reaction, graphene was centrifuged from the reaction mixture and washed with water and ethanol repeatedly six times. Finally, graphene was dried in a vacuum oven for 3 h at 70°C.

**Synthesis of Co$_3$S$_4$/G:** Co$_3$S$_4$ was synthesized by using 1D sacrificial wires of Co(CO)$_{0.35}$Cl$_{0.20}$(OH)$_{1.10}$ as a template for the growth of Co$_3$S$_4$ NTs, which were prepared by mixing a solution of CoCl$_2$·6H$_2$O (2.5 mmol) and Co(NH$_3$)$_2$ (2.5 mmol) in water (20 mL). The mixture was stirred and then transferred to a 25 mL Teflon-lined stainless-steel autoclave, which was placed in an oven at 100°C for 12 h. After completion of the reaction, the product was collected by centrifugation and washed six times with copious amounts of water and ethanol. Finally, the solid product was dried at 70°C for 5 h in a vacuum oven. The composites were prepared by using a 1:0.70 (w/w) ratio of Co(CO)$_{3.5}$Cl$_{2.0}$(OH)$_{1.10}$ and graphene, respectively. The ratio between Co(CO)$_{3.5}$Cl$_{2.0}$(OH)$_{1.10}$ and thioacetamide (sulfur source) was maintained at 1:2. Initially, Co(CO)$_{3.5}$Cl$_{2.0}$(OH)$_{1.10}$ and the graphene were sonicated separately for 30 min in water (20 mL) and then transferred to a 50 mL Teflon-lined stainless-steel autoclave with thioacetamide. After stirring for a few minutes, the reaction mixture was aged at 200°C for 12 h in an autoclave. After completion of the reaction, the product was centrifuged, then washed six times with water and ethanol, and then dried at 70°C for 5 h under vacuum. A sample of Co$_3$S$_4$ was also prepared in the absence of graphene to observe the effect of the latter on the properties of Co$_3$S$_4$.

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