

Synthesis of Cu_2O /reduced graphene oxide composites as anode materials for lithium ion batteries

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Abstract: A facile way was used to synthesize Cu_2O /reduced graphene oxide (rGO) composites with octahedron-like morphology in aqueous solution without any surfactant. TEM images of the obtained Cu_2O /rGOs reveal that the Cu_2O particles and rGO distribute hierarchically and the primary Cu_2O particles are encapsulated well in the graphene nanosheets. The electrochemical performance of Cu_2O /rGOs is enhanced compared with bare Cu_2O when they are employed as anode materials for lithium ion batteries. The Cu_2O /rGO composites maintain a reversible capacity of 348.4 mA·h/g after 50 cycles at a current density of 100 mA/g. In addition, the composites retain 305.8 mA·h/g after 60 cycles at various current densities of 50, 100, 200, 400 and 800 mA/g.

Key words: cuprous oxide; reduced graphene oxide; anode material

1 Introduction

Currently, transition metal oxides, such as Co_3O_4 , Fe_2O_3 , Mn_3O_4 and CuO , have attracted great research interests as alternative anode materials for lithium ion batteries because of their low cost and relatively high specific capacity compared with graphite materials [1–8]. However, these conversion-based materials have inherently inferior cycling performance due to their severe volume expansion and contraction during the discharge and charge process. Cuprous oxide (Cu_2O) is also investigated as an anode material for lithium ion batteries, while it suffers from the same drawback of other transition metal oxides. Previous studies have confirmed that different particle size and morphology play a significant role in determining the electrochemical performance of Cu_2O crystal [9–12].

Graphene was confirmed an excellent supporting material as it owns high surface area, superior electrical conductivity and flexibility [13,14]. XU et al [15] fabricated cubic-like cuprous oxide/graphene by two steps in absolute ethanol. Besides, it has been reported that Cu_2O /reduced graphene oxide (rGO) composites were synthesized by one-pot hydrothermal treatment or by high intensity ultrasound assisting [16–19]. However,

these methods are relatively complicated and are difficult to scale up. Moreover, graphene can disperse in water directly without any assistance of dispersing agent [20]. In order to buffer the volume change and alleviate the aggregation of graphene during the cycling process, constructing Cu_2O /rGO composites is efficient to enhance the cycling performance and rate capability of Cu_2O .

In this work, a facile way to synthesize Cu_2O /rGO composites with octahedron-like morphology composites is represented. The composites are prepared by the reduction of GO and $\text{Cu}(\text{OH})_4^{2-}$ simultaneously using hydrazine as reducer in aqueous solution under a mild temperature and constant pressure without any surfactant.

2 Experimental

2.1 Preparation of Cu_2O and Cu_2O /rGO

Graphene oxide (GO) was synthesized from natural graphite by modified Hummer's method [21]. To prepare Cu_2O /rGO, 200 mL aqueous solution of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (2.5 g) was added into round-bottomed flask, followed by 200 mL aqueous solution of NaOH (1.6 g) under magnetic stirring. After that, 100 mL GO (1 mg/mL) aqueous that was dispersed by sonication for 1 h was

mixed in. Once the temperature of the mixed solution reached 50 °C, 3 mL hydrazine solution (1.5 mol/L) was added into it. After 2 h, the brown solid products were collected by filtration, washed with deionized water and ethanol three times, respectively, and dried in a vacuum oven at 80 °C for 12 h. The bare Cu_2O was prepared by the same process without adding GO solution.

2.2 Materials characterization

The crystal structure of the products was determined by X-ray diffraction (XRD, Cu K_α radiation, Rint-2000, Rigaku). The morphology of the products was observed by scanning electron microscopy (SEM, Sirion200) and transmission electron microscopy (TEM, Tecnai G12). Fourier transform infrared (FT-IR) spectra were recorded by a Nicolet AVATAR 360 FTIR spectrometer. The content of graphene was determined by a carbon sulfur analyzer (HCS-140, Shanghai Dekai).

2.3 Electrochemical measurement

Electrochemical performance was measured in CR2025 coin-type cell with a Neware galvanostatic charge–discharge system. The anode electrodes were prepared by coating slurries that were made up of Cu_2O or $\text{Cu}_2\text{O}/\text{rGO}$ (80% in mass fraction) with acetylene black (10% in mass fraction) and poly(vinylidene fluoride) (10% in mass fraction) as a binder dissolved in N-methyl pyrrolidinone solution on a copper foil. Lithium foil was used as a counter electrode. A polypropylene micro-porous film was employed as the separator. The electrolyte was a mixture of 1 mol/L LiPF_6 in a mixture of ethylene carbonate (EC), ethyl methyl carbonate (EMC) and dimethyl carbonate (DMC) (1:1:1, v/v/v). The batteries were cycled galvanostatically between 0.001 V and 3.0 V at room temperature. Cyclic voltammetry (CV) test was carried out in a CHI600d electrochemical workstation at a scanning rate of 0.1 mV/s from 0 to 3.0 V. Electrochemical impedance spectroscopy (EIS) measurements were conducted with a CHI600d potentiostat over a frequency range from 100 kHz to 10 mHz. A three-electrode cell in a discharged-state was used for EIS test, where lithium foils were used as both reference and counter electrode.

3 Results and discussion

3.1 Materials characterization

To hinder the aggregation between the electronegative functional groups of GO and electropositive cupric ions caused by electrostatic attraction, the NaOH solution was added into CuSO_4 solution according to the stoichiometric ratio to form the coordination compound $\text{Cu}(\text{OH})_4^{2-}$. Therefore, the GO and $\text{Cu}(\text{OH})_4^{2-}$ were dispersed homogeneously in

aqueous [22]. They were reduced simultaneously once the hydrazine solution was added, and the $\text{Cu}_2\text{O}/\text{rGO}$ composites were prepared when the amount of hydrazine was appropriate. Despite our synthetic approach is different from the method of previous studies to synthesize metal oxides/rGO, in which the metal ions were added into the GO suspension and coordinated with carboxyl groups of GO [2,3,15–19,23,24], the newly formed Cu_2O particle will be embedded well in the reduced graphene oxide either.

Figure 1(a) shows the X-ray diffraction patterns of $\text{Cu}_2\text{O}/\text{rGO}$ composites, pure graphene and GO. The GO shows a sharp (001) diffraction peak, which indicates that the interplanar spacing of GO is 0.840 nm. In addition, the pure graphene displays a broad (002) diffraction peak, which implies the interplanar spacing of rGO is 0.359 nm. Consequently, the decreased interplanar spacing demonstrates that the amount of GO reduces. The Bragg peaks of the Cu_2O are clearly distinguishable, and all peaks can be exclusively indexed to the cubic phase Cu_2O (JCPDS file No.05–0667), indicating the good crystalline nature of the Cu_2O particle. It should be noted that no diffraction peaks for graphite could be observed, which suggests no further agglomeration of few layers of rGO in $\text{Cu}_2\text{O}/\text{rGO}$ composites. The graphene content in the composites

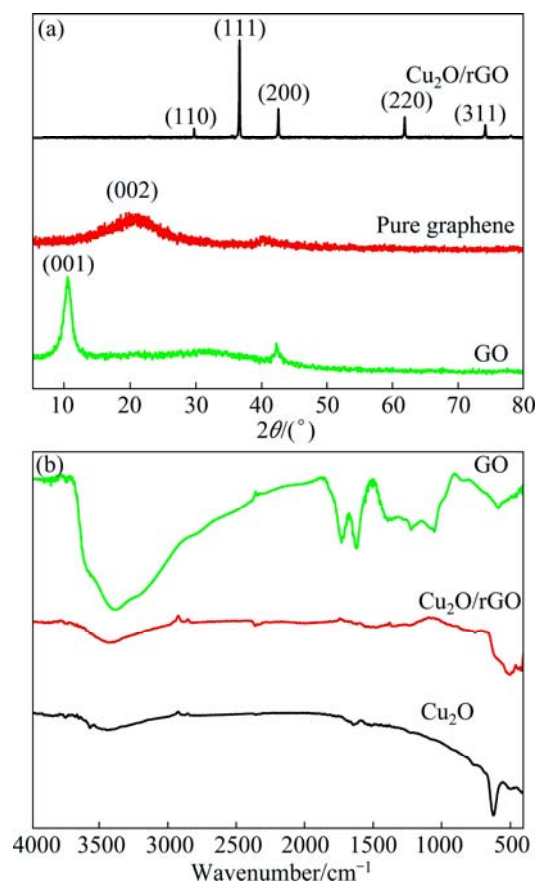


Fig. 1 XRD patterns of $\text{Cu}_2\text{O}/\text{rGO}$, pure graphene and GO (a) and FTIR spectra of GO, $\text{Cu}_2\text{O}/\text{rGO}$ and Cu_2O (b)

is 5.455% that was determined by a carbon sulfur analyzer, which means the constituent of Cu_2O in the composites is 94.545%. FTIR spectra of GO, Cu_2O , and $\text{Cu}_2\text{O}/\text{rGO}$ are illustrated in Fig. 1(b). GO displays plentiful oxygen-containing groups, and the band around 3400 cm^{-1} arises from stretching vibration of O—H. The band at 1720 cm^{-1} reflects the C=O vibration of —COOH located at the edge of GO, and the peaks around 1367 cm^{-1} , 1225 cm^{-1} and 1056 cm^{-1} can be attributed to the C—O groups on the surface of GO. While for Cu_2O , the peak around 622 cm^{-1} is ascribed to Cu—O vibrating, and the relatively weak peaks at 3428 cm^{-1} are caused by the hydroxy of absorbed water inevitably. As for $\text{Cu}_2\text{O}/\text{rGO}$, the Cu—O vibration shifts to 506 cm^{-1} , suggesting that the interaction between graphene and Cu_2O affects the vibration of Cu—O bonds.

The scanning electron microscopy images shown in Figs. 2(a) and (b) represent the typical Cu_2O and $\text{Cu}_2\text{O}/\text{rGO}$ crystals synthesized, respectively. The Cu_2O crystals with octahedron morphology are clearly seen with sizes ranging from 0.5 to $1\text{ }\mu\text{m}$. However, the characteristic of octahedron morphology in $\text{Cu}_2\text{O}/\text{rGO}$ crystals is not as evident as Cu_2O . This may be caused by the existence of graphene that affects the growth of Cu_2O crystals. In addition, rGO sheets in $\text{Cu}_2\text{O}/\text{rGO}$ enwrap the Cu_2O crystals and the size of $\text{Cu}_2\text{O}/\text{rGO}$ is around

400 nm , which can be clearly seen in Fig. 2(c). The high magnification TEM image of the edge in Fig. 2(d) reveals that the Cu_2O particles and rGO distribute hierarchically.

3.2 Electrochemical properties

In order to evaluate the electrochemical performance of bare Cu_2O and $\text{Cu}_2\text{O}/\text{rGO}$, galvanostatic discharge–charge experiments were carried out. The 1st, 5th, 10th discharge–charge curves at a constant current density of 50 mA/g are illustrated in Figs. 3(a) and (b), in which the potential range is settled from 0.001 to 3.0 V , and the inset in Fig. 3(a) is the 1st discharge–charge curve of rGO. In the first cycle, the bare Cu_2O shows a discharge and charge capacity of $667.7\text{ mA}\cdot\text{h/g}$ and $330.8\text{ mA}\cdot\text{h/g}$ respectively, and the initial coulombic efficiency is as low as 49.5%. In comparison, the $\text{Cu}_2\text{O}/\text{rGO}$ composites deliver a discharge and charge capacity of 771.7 and $567.3\text{ mA}\cdot\text{h/g}$ respectively, which corresponds to 73.6% of the coulombic efficiency. The increased capacity ($104\text{ mA}\cdot\text{h/g}$) is attributed to the addition of rGO because the rGO can delivery $1556.8\text{ mA}\cdot\text{h/g}$ in the first discharge as shown in the inset of Fig. 3(a). Moreover, the reason why the initial coulombic efficiency rises in $\text{Cu}_2\text{O}/\text{rGO}$ composites is that the rGO makes the conversion reaction of Cu_2O proceed easier. Furthermore, for bare Cu_2O , the reversible capacity

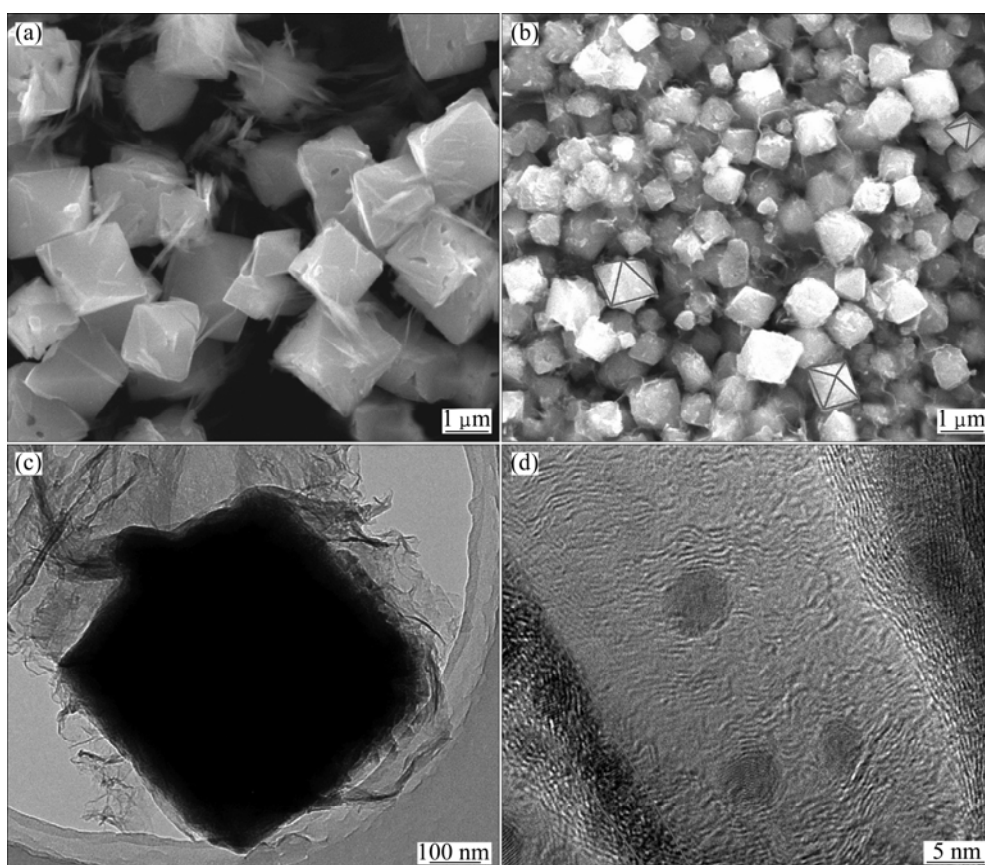


Fig. 2 SEM images of Cu_2O (a) and $\text{Cu}_2\text{O}/\text{rGO}$ (b), TEM image of $\text{Cu}_2\text{O}/\text{rGO}$ (c) and HRTEM image of $\text{Cu}_2\text{O}/\text{rGO}$ in edge (d)

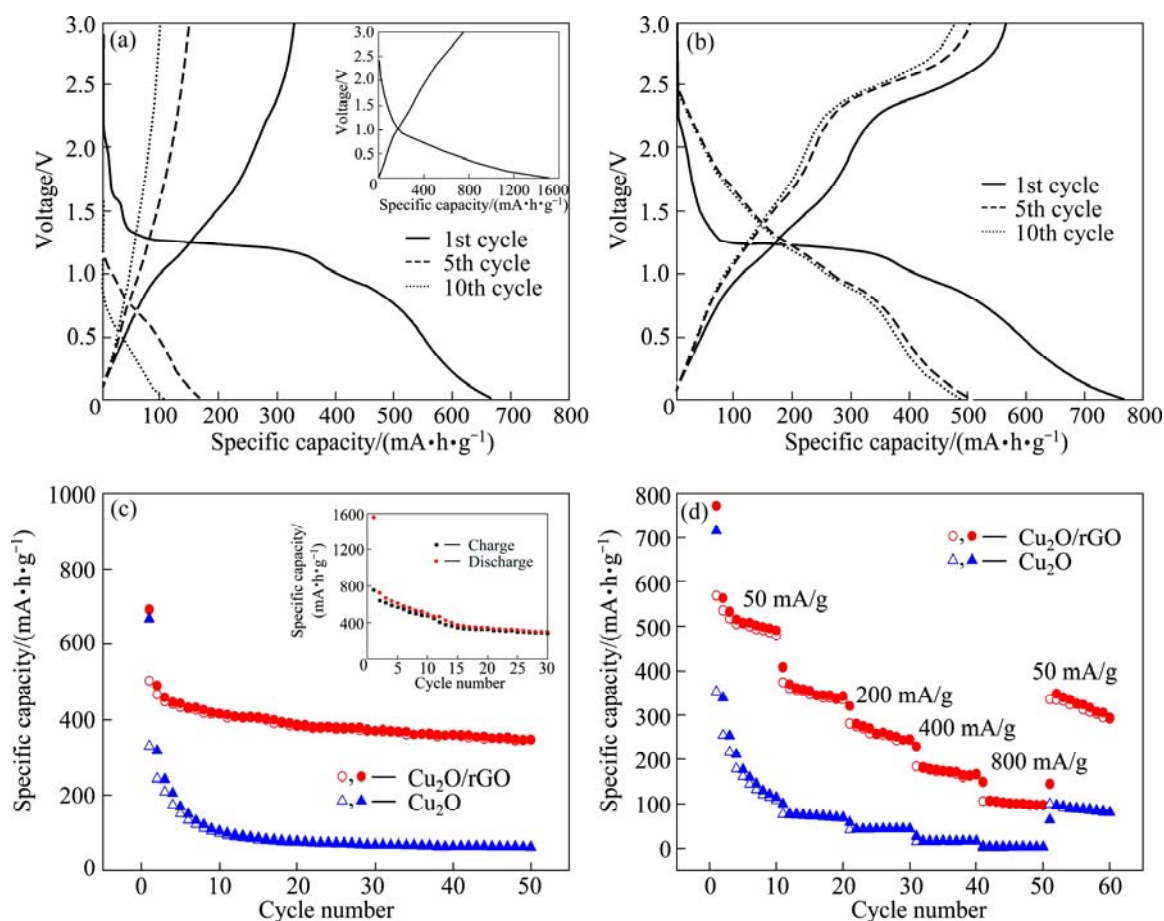


Fig. 3 Discharge and charge curves of bare Cu_2O at 50 mA/g (inset shows the first discharge and charge curves of rGO at 50 mA/g) (a), $\text{Cu}_2\text{O}/\text{rGO}$ at 50 mA/g (b), cycling performance at 50 mA/g (inset shows cycling performance of rGO at 50 mA/g) (c) and rate capability at various current densities (d)

drops to 105.8 mA·h/g, whereas $\text{Cu}_2\text{O}/\text{rGO}$ composites still exhibit 478.6 mA·h/g after 10 cycles.

Figure 3(c) demonstrates the cycling performance of bare Cu_2O and $\text{Cu}_2\text{O}/\text{rGO}$ at a current density of 50 mA/g. As for bare Cu_2O , the reversible capacity drops to 64.1 mA·h/g after 50 cycles while $\text{Cu}_2\text{O}/\text{rGO}$ still retains 348.4 mA·h/g, which demonstrates that the cycling capability is improved significantly. The poor capacity retention ability of bare Cu_2O should be caused by the large volume exchange during the cycling process. In addition, the reversible capacity of bare rGO remains 301.6 mA·h/g after 30 cycles because rGO is prone to stack during the cycling process [14]. Figure 3(d) represents the rate capability of bare Cu_2O and $\text{Cu}_2\text{O}/\text{rGO}$ from current densities of 50 to 800 mA/g after 10 cycles at each current density. After 60 cycles at various current densities, the $\text{Cu}_2\text{O}/\text{rGO}$ composites maintain a reversible capacity of 305.8 mA·h/g. Even at a current density of 800 mA/g, the reversible capacity of $\text{Cu}_2\text{O}/\text{rGO}$ composites is higher than that of bare Cu_2O at a current density of 50 mA/g. These electrochemical data indicate that a little amount of graphene (5.455%) has a beneficial effect on the cycling performance and rate

capability. This can be ascribed to two main reasons. The first is that the addition of rGO reduces the size of Cu_2O , which is considered an effective way to improve the electrochemical performance of Cu_2O as anode material for lithium ion battery. The other is that the superior flexibility of rGO is beneficial to buffering the huge volume exchange of Cu_2O during cycling process.

To further investigate the electrochemical performance, CV experiments were conducted. The cyclic voltammetric curves of bare Cu_2O and $\text{Cu}_2\text{O}/\text{rGO}$ between 0 and 3 V at a scanning rate of 0.1 mV/s are shown in Figs. 4(a) and (b), respectively. For bare Cu_2O sample in the first cycle, there is a well-defined peak ranging from 1.5 to 0.5 V, and a broad peak in the range of 0.4–0 V is observed during the cathodic process. The former peak can be attributed to the reduction of Cu_2O to Cu and formation of Li_2O , and the latter can be ascribed to the formation of a solid electrolyte interface (SEI) film [9]. As for the anodic process, two peaks in a range of 1.0–1.8 V are due to the decomposition of the SEI film, and the peak around 2.4 V corresponds to the conversion of Cu to Cu_2O and the decomposition of Li_2O . In the second cycle, the cathodic peak around 1.0 V in the first

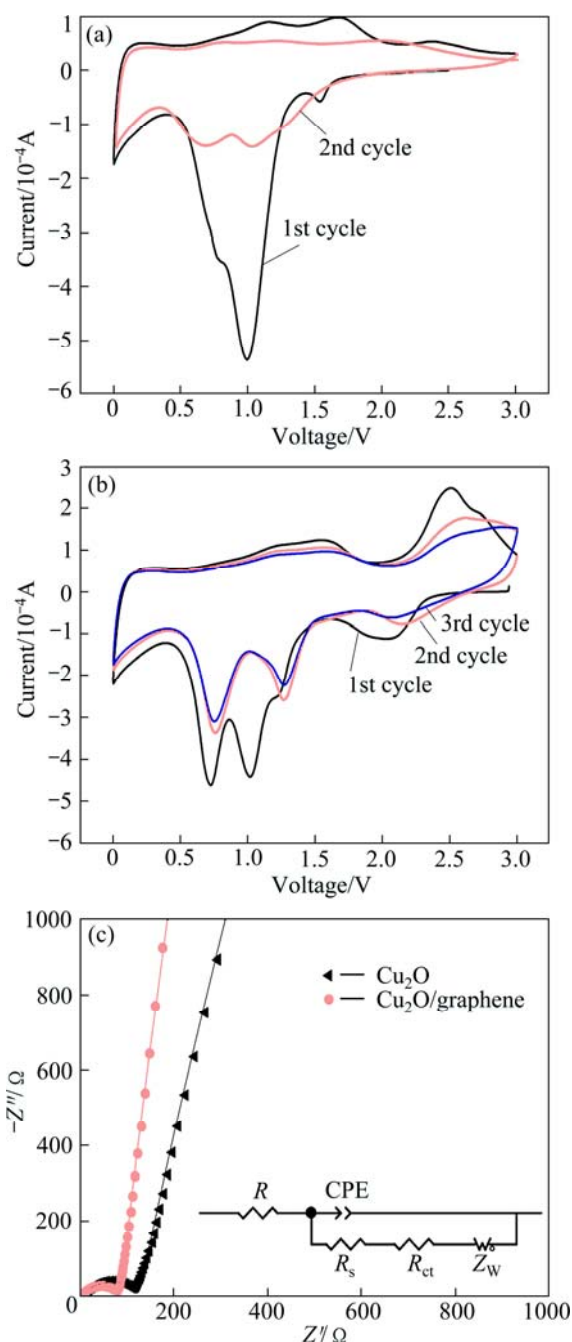


Fig. 4 Cyclic voltammograms of bare Cu_2O with scan rate of 0.1 mV/s between 0 and 3.0 V (vs Li/Li^+) (a), $\text{Cu}_2\text{O}/\text{rGO}$ (b), and Nyquist plots of bare Cu_2O and $\text{Cu}_2\text{O}/\text{rGO}$ (c) (The solid symbols denote experimental data and the lines represent the fitting results based on equivalent circuit (inset in (c)))

cycle is separated to two cathodic peaks. This owes to the decreased particle size of Cu_2O , which leads to the potential of the conversion reaction between Cu_2O and Li^+ increasing [10]. Compared with bare Cu_2O , there is a cathodic peak positioned at about 2.0 V, which indicates that Li^+ inserts into graphene and is consistent with the initial discharge curve of pure graphene (inset in Fig. 3(a)). In addition, the peak between 1.5 to 0.5 V is divided into two peaks in the first cathodic process rather

than in the second cathodic process, which is correlated well with the TEM analysis that the size of Cu_2O is decreased in $\text{Cu}_2\text{O}/\text{rGO}$ composites [10]. Moreover, the peaks around 2.5 V are stronger and sharper, indicating that the conversion reaction between Cu_2O and Li^+ occurs more easily. Furthermore, the cyclic voltammogram curve of the second cycle overlaps well with that of the third cycle, suggesting that the cycling performance of $\text{Cu}_2\text{O}/\text{rGO}$ is enhanced.

For the sake of studying the function of graphene further, EIS measurements were performed. Nyquist plots of Cu_2O and $\text{Cu}_2\text{O}/\text{rGO}$ electrode at open circuit voltage are presented in Fig. 4(c). Generally, it is believed that the semicircle in the medium-frequency region is assigned to the charge-transfer resistance (R_{ct}), and the line in the lower frequency region corresponds to the Warburg impedance. It can be seen apparently that the semicircle in the medium frequency of $\text{Cu}_2\text{O}/\text{rGO}$ is smaller than that of Cu_2O . According to the fitting results based on the experimental data using equivalent circuit (inset in Fig. 4(c)), the R_{ct} is decreased from 128.01 to 84.07 Ω . The result is consistent with the electrochemical data and analysis from Fig. 3(d).

4 Conclusions

1) A facile way to synthesize $\text{Cu}_2\text{O}/\text{rGO}$ composites in aqueous solution without any surfactant instead of the commonly hydrothermal process to prepare metal oxide/rGO is developed.

2) The as-synthesized $\text{Cu}_2\text{O}/\text{rGO}$ composites are hierarchical in microscopic structure. The Cu_2O particles cross-link well with rGO and show better cycling performance and rate capability than bare Cu_2O when they are employed as anode materials for lithium ion batteries.

3) The existing of rGO can buffer the volume expansion and concentration as a flexible constraint and improve the conductivity of $\text{Cu}_2\text{O}/\text{rGO}$ composites.

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锂离子电池用氧化亚铜/石墨烯负极材料的制备

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摘 要: 在不添加表面活性剂的水溶液体系中, 采用水合肼作为还原剂制备得到具有八面体形貌的氧化亚铜/石墨烯复合材料。透射电镜分析表明: 氧化亚铜颗粒与石墨烯在复合物中呈多层次分布, 而且氧化亚铜一次颗粒很好地嵌入在石墨烯层间。相比于纯氧化亚铜, 氧化亚铜/石墨烯复合材料作为锂离子电池负极材料的电化学性能得到了显著的改善。在 100 mA/g 的电流密度下循环 50 次后, 氧化亚铜/石墨烯复合物的可逆比容量高达 348.4 mA·h/g, 同时, 在不同倍率下(50, 100, 200, 400, 800 mA/g)循环 60 次后, 其可恢复容量仍达 305.8 mA·h/g。

关键词: 氧化亚铜; 还原氧化石墨烯; 负极材料

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