



FeS anchored reduced graphene oxide nanosheets as advanced anode material with superior high-rate performance for alkaline secondary batteries



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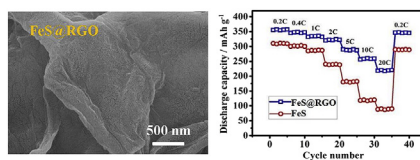
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HIGHLIGHTS

- For the first time, FeS@RGO nanosheets are synthesized via a facile approach.
- FeS@RGO is firstly used as anode materials for alkaline rechargeable batteries.
- FeS@RGO exhibits superior high-rate charge/discharge capability and cycle stability.
- The enhanced performance mainly results from the robust sheet-anchored structure.

GRAPHICAL ABSTRACT



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ABSTRACT

A new nanocomposite formulation of the iron-based anode for alkaline secondary batteries is proposed. For the first time, FeS nanoparticles anchored on reduced graphene oxide (RGO) nanosheets are synthesized via a facile, environmentally friendly direct-precipitation approach. In this nanocomposite, FeS nanoparticles are anchored uniformly and tightly on the surface of RGO nanosheets. As an alkaline battery anode, the FeS@RGO electrode delivers a superior high-rate charge/discharge capability and outstanding cycling stability, even at a condition without any conductive additives and a high electrode loading of $\sim 40 \text{ mg cm}^{-2}$. At high charge/discharge rates of 5C, 10C and 20C (6000 mA g^{-1}), the FeS@RGO electrode presents a specific capacity of ~ 288 , 258 and 220 mAh g^{-1} , respectively. Moreover, the FeS@RGO electrode exhibits an admirable long cycling stability with a superior capacity retention of 87.6% for 300 cycles at a charge/discharge rate of 2C. The excellent electrochemical properties of the FeS@RGO electrode can be stemmed from the high specific surface area, peculiar electric conductivity and robust sheet-anchored structure of the FeS@RGO nanocomposite. By virtue of its superior fast charge/discharge properties, the FeS@RGO nanocomposite is suitable as an advanced anode material for high-performance alkaline secondary batteries.

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1. Introduction

Owing to their scalability, high energy efficiency, eco-friendliness and flexibility, rechargeable batteries are particularly suitable for powering hybrid electric vehicles (HEVs) and all-electric vehicles (EVs) [1–5]. Thus far, lithium ion batteries (LIBs), lead-acid batteries and nickel-based alkaline rechargeable batteries have been widely used in various applications. However, the toxicity of lead-acid and Ni–Cd batteries, which present serious ecological threats, is seemingly an insurmountable problem [6,7]. The further development of Ni-MH batteries is limited by their high cost of the MH anode material. In addition to low cost and eco-friendliness, safety consideration has attracted significant attention to power rechargeable batteries. As is known, the safety issues of non-aqueous LIBs cannot be ignored due to the use of highly toxic and flammable organic electrolytes [2,4]. Besides, there is a high cost of the complicated process for assembling non-aqueous LIBs due to the strict atmosphere requirements. As such, many types of aqueous rechargeable nickel/metal batteries, such as Ni/Fe batteries [6,8–15], Ni/Zn batteries [16–19] and Ni/Co batteries [20–24], have attracted considerable attention in recent years because of their good safety performance, high ionic conductivity, little environmental issues, and low cost.

Among various nickel/metal batteries, aqueous Ni/Fe batteries are particularly favorable due to the following advantages. In contrast with the Zn anode, Fe-based anode materials are insoluble in alkaline solutions, leading to a long cycle life [25–27]. Compared with the Co anode, iron-based materials are globally abundant, non-toxic, and cost effective [27]. Indeed, the Ni/Fe battery was invented in 1899–1902 and has been well used in many fields [27,28]. However, conventional iron electrodes using Fe₃O₄ or Fe materials always suffer from the intrinsic drawbacks of poor discharge rate capability resulting from their passivation behavior and relatively low charging efficiency caused by considerable hydrogen evolution [12,13,25,29].

Considerable work has been conducted to overcome the aforementioned issues of iron electrodes. Various approaches include using novel and nano-sized materials [8,11,14,30–34], anode additives [15,35–39], and electrolyte additives [12,13,40]. For example, the performance of Fe₂P nanoparticles as a new anode active material for the iron electrode has been reported with a high discharge capacity of 413 mAh g⁻¹ but a low charging efficiency of 68.8% [34]. Two types of FeO_x-graphene hybrid with a high-rate charge/discharge ability or high discharge capacity have successfully been synthesized and applied as anode materials for nickel-iron batteries [7,10]. Manohar et al. [11,12,15,41] have developed a high-performance iron electrode using carbonyl iron as the anode material. These carbonyl iron electrodes deliver a high charging efficiency of 92% and excellent rate performance. Recently, we have succeeded in synthesizing FeS/C composites as a novel anode material for Ni/Fe batteries using a simple calcination method followed by a co-precipitation process [8]. It was found that the FeS/C composite is highly desirable for Ni/Fe rechargeable batteries. Due to its excellent anti-corrosion property in the alkaline electrolyte, the as-prepared FeS/C composite exhibits attractive rate capability with a specific capacity of nearly 230 mAh g⁻¹ at an impressive 5C rate (1500 mA g⁻¹). Although considerable progresses have been made, the high-rate performance of the iron electrodes still needs to be substantially improved. As such, the objective of this work is to further explore the novel FeS-based anode material to meet the requirements of HEVs and EVs where elevated current response is needed.

Since the discovery of graphene in 2004 [42], it has attracted

tremendous scientific interests in light of its intriguing characteristics, such as unique two-dimensional robust nanostructure, high specific surface area, and excellent electronic conductivity. Up to now, Graphene-based composites have been extensively explored in a wide range of applications [43]. A large numbers of reports have demonstrated that graphene or reduced graphene oxide (RGO) can act as not only an excellent host for loading active materials but also a promising conductive material for electrodes to improve their electrochemical performance [7,43–47]. Recently, Fei et al. [48] produced a RGO wrapped FeS nanocomposite as the lithium-ion battery anode material via a facile direct-precipitation method. Owing to the robust sheet-wrapped structure, the resulting nanocomposites exhibit better electrochemical performance than FeS prepared without RGO sheets. As stated above, we have proved that micro-sized FeS and FeS/C composites can deliver excellent high rate and cyclic performance due to their unique inherent characteristics. Following upon our previous work, reducing the particle size of FeS to nanoscale and anchoring nano-sized FeS on the surface of RGO nano-sheet may be promising to further enhance high-rate performance of the FeS anode for Ni/Fe batteries. However, to the best of knowledge, the application of the FeS@RGO nanocomposite as an anode material in alkaline secondary batteries has not been reported so far.

In this context, we report a facile direct-precipitation strategy to synthesize the FeS@RGO nanocomposite. As expected, due to their advantageous nanostructure, the FeS@RGO nanosheets display unprecedented high capacities with stable cycling performances at fast charge/discharge rates (2C–20C). For example, the as-prepared FeS@RGO nanocomposite electrode with a high loading of ~40 mg cm⁻² and without any conductive additives demonstrates a high reversible capacity of ~220 mAh g⁻¹ at a charge-discharge rate of 20C (6000 mA g⁻¹). To the best of our knowledge, such a superior FeS/RGO nanocomposite for an alkaline iron electrode has never been reported.

2. Experimental

2.1. Synthesis of FeS and the FeS/RGO nanocomposite

Graphene oxide (GO) was synthesized by a modified Hummers method using natural graphite [49]. The Raman spectra and XRD pattern of the as-prepared GO precursor were illustrated in Fig. S1 (supporting information), which are in accordance with the previous report [49]. The FeS@RGO nanocomposite was prepared by a simple direct-precipitation. A typical synthesis procedure of the FeS@RGO nanocomposites schematically illustrated in Fig. 1a. Firstly, the GO suspension was dispersed in FeSO₄ solution by stirring. After ultra-sonication, Fe²⁺ ions could easily adsorb onto the GO surface through oxygen-containing functional groups (e.g., –OH, –COOH). Then the introduction of Na₂S led to a uniform FeS coating on the surface of the GO nanosheets. After that, the as-prepared black products were annealed in an Ar atmosphere to form the final FeS@RGO nanocomposite.

Typically, 3 g of FeSO₄·7H₂O was dissolved in 150 mL deionized water, and 10 ml of the as-prepared GO (8 mg/ml) was added to the resulting solutions under vigorous stirring to form a mixture, followed by continuous stirring for 1 h. The mixture was then sonicated for 1 h. 3.1 g of Na₂S·9H₂O was dissolved in 30 ml deionized water and then the fresh Na₂S solution was dropwise added into the above mixture under constant mechanical stirring in a N₂ atmosphere at room temperature, followed by vigorous stirring for 1 h. Finally, a solid product was filtrated, washed several times with deionized water, dried at 80 °C for 12 h in a vacuum environment

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