Fabrication of multifunctional carbon encapsulated Ni@NiO nanocomposites for oxygen reduction, oxygen evolution and lithium-ion battery anode materials

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ABSTRACT Multifunctional carbon encapsulated Ni@NiO nanocomposites (Ni@NiO@C) were synthesized for applications in oxygen reduction reactions (ORR), oxygen evolution reactions (OER) and lithium-ion batteries (LIB). The morphology was investigated *via* SEM and TEM, suggesting that the Ni@NiO@C nanocomposites have uniform and spherical core-shell structures. When the Ni@NiO@C nanocomposite is used as the catalyst in ORR, 90% of the initial current density can be maintained after 15 h in O₂-saturated 0.1 mol L⁻¹ KOH at 0.3 V under a rotation speed of 1600 rpm. As a catalyst for OER, the highest activity overpotential of the Ni@NiO@C nanocomposite electrocatalyst is 380 mV (*vs.* RHE) under the current density of 10 mA cm⁻², and the Tafel slope was calculated to be 55 mV dec⁻¹ by linear fitting. Electrochemical performances of the Ni@NiO@C nanocomposites used as LIB electrodes exhibited a long cycling life with a high capacity of 750 mA h g⁻¹ after 400 cycles under 200 mA g⁻¹.

Keywords: oxygen evolution reaction, lithium-ion battery, oxygen reduction reaction, NiO

INTRODUCTION

With the development of science and technology in society, the energy crisis and environmental pollution are becoming increasingly acute [1]. The energy crisis will affect the human lifestyle deeply by affecting energy access, consumption, and storage [2]. Therefore, the replacement of expensive and rare metal catalysts is a critical issue in industrial and academic fields. To minimize the effects, widespread investigations have been performed, taking both experimental and theoretical

viewpoints [3]. The oxygen reduction reaction (ORR), with favorable activity and stability, was one of the key factors for the development of renewable energy in fuel cells and batteries [4,5]. One possible way to obtain clean energy is to produce hydrogen from water splitting. In this procedure, oxygen evolution reaction (OER) performance with low overpotential and high current densities is very important for large-scale production [6]. Considering energy storage [7], the lithium-ion battery (LIB) has been extensively studied because of its excellent properties, including large capacity and long-life cycling. The development of LIB anode materials with high capacities and super-long cyclic stability is one of the most important tasks [8]. Lately, nanocomposites based on various carbon materials [9] are being widely studied by researchers to resolve energy and environmental problems [10,11]. Carbon nanotubes, nanospheres, graphene nanocomposites, and so on have been studied as ORR electrocatalysts, OER electrocatalysts, and LIB anodes with excellent performances [12–14]. It would be a great challenge as well as huge progress for practical applications to combine these advantages in one carbon-based nanocomposite [15].

To achieve this, a possible solution is decorating the surface of the carbon substrate with metal and metal oxides [16]. Because of advantages such as high OER catalytic activity [17], high capacity as LIB anodes [18], and high activity and stability as ORR electrocatalysts, nickel and nickel oxides have been widely investigated as candidates for decorating carbon substrates [4]. As we know, improving the stability of ORR, enhancing the

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Figure 1 Schematic illustration of the formation of Ni@NiO@C nanocomposites.

conduction of OER, and reducing the pulverization effect in LIB are the keys to improve the electrochemical performances of nanocomposites, which can be severely restricted by the architectures of the different components of the nanocomposites [19]. Therefore, it is important to further investigate composites of nickel and nickel oxides and carbon materials for multifunctional applications.

In this paper, multifunctional carbon-encapsulated Ni@NiO nanocomposites (Ni@NiO@C) were synthesized and investigated systematically. Improvements in stability during ORR, enhancements in conduction in OER, and reduction of the pulverization effect in LIB were achieved, leading to excellent Ni@NiO@C performances in ORR, OER, and LIB.

EXPERIMENTAL SECTION

A precursor solution was prepared by dissolving Ni(NO₃)₂·6H₂O, glucose, and sodium chloride in deionized water with stirring for 10 min. The precursor solution was stirred and boiled in a hot water bath at 80°C. Then, the intermediate product Ni(NO₃)₂·C₆H₁₂O₆ coated with NaCl was calcined at 750°C for 2 h under an argon atmosphere (50 mL min⁻¹) and then annealed at 300°C for 4 h in air. After cooling to room temperature, the obtained powder was washed with deionized water to dissolve the sodium chloride. Finally, the Ni@NiO@C nanocomposites were synthesized (as shown in Fig. 1).

The morphology and nanostructure of Ni@NiO@C nanocomposites were characterized by scanning electron microscopy (SEM, S-4800 Hitachi, Japan) and transmission electron microscopy (TEM, JEM-2010 JEOL, Japan). The crystal structure of the nanocomposite was measured

using a Rigaku SmartLab X-ray diffractometer with Cu-Kα radiation ($\lambda = 1.5406 \text{ \AA}$). The Raman spectra were characterized at room temperature by a Renishaw inVia micro Raman spectroscope with a laser radiation of 514 nm. The surface area and porous structure were determined by nitrogen adsorption at 77 K with a Quantachrome Autosorb, and the pore size distributions were obtained from nitrogen adsorption isotherms.

The Ni@NiO@C nanocomposites (50 mg) were dispersed in 10 mL mixed liquid of nafion (100 μL , 5 wt.%), ethanol and distilled water (1:4 v/v), and sonicated for about 10 min to obtain a homogeneous solution. Then, 10 μL of the solution was drawn out and loaded onto a glassy carbon rotating-disk electrode (RRDE-3A RRDE, ALS Co., Ltd., Japan), which has a standard three-electrode cell including platinum mesh as the auxiliary electrode. Linear scan voltammetry (LSV) and chronoamperometric response measurements were performed on rotating-disk electrode (RDE) in a 1 mol L⁻¹ KOH electrolyte solution by using a Chenhua electrochemical workstation (CHI760E, China).

Using *N*-methyl pyrrolidinone (NMP) as a solvent, the Ni@NiO@C nanocomposites were mixed with both super carbon black and a polymer binder made of polyvinylidene fluoride (PVDF) with a weight ratio of 80:10:10, resulting in a black slurry. The electrode was prepared by coating the slurry onto copper foil and dried at 80°C for 12 h in a vacuum oven. The electrolyte solution was 1 mol L⁻¹ LiPF₆ in a mixture of ethylene carbonate and diethyl carbonate with a volume ratio of 1:1 and a polyethylene separator (Celgard 2400) was used as the separator. The lithium foil was used as the cathode of the

coin cell. 2032-type coin cells were assembled in an Ar-filled glove box (Mikrouna, super 1220/750/900), with both moisture and oxygen concentrations below 0.01 ppm. Cyclic voltammetry (CV) and galvanostatic charge/discharge measurements were performed on a Chenhua electrochemical workstation (CHI660E, China) and a LAND galvanostatic charge-discharge instrument (CT2001A, China).

RESULTS AND DISCUSSION

The morphology of Ni@NiO/C nanocomposites was characterized using SEM and TEM, as shown in Fig. 2a–c. The Ni@NiO/C nanocrystals show a very uniform spherical shape with a diameter of 50–100 nm. Although they are overlapping, the Ni@NiO/C nanocomposites are separated by the C shell without agglomeration. In Fig. 2c, the Ni@NiO/C nanocomposites' core-shell structure can be clearly seen from the high-magnification TEM image. The dark-colored core is Ni@NiO, and the C shell is flocculent and almost transparent. As shown in the high-resolution TEM image (Fig. 2d), the interplanar spacing is measured to be 0.20 nm, which corresponds to the (111) crystal plane of face center cubic structure Ni. The NiO shell is confirmed by the X-ray diffraction (XRD) pattern shown in Fig. 3a. The diffraction peaks of NiO located at 37.2°, 43.3°, and 62.8° are detected, and correspond to the (311), (331), and (440) crystal planes of NiO, respectively. Lastly, the C component is verified by the Raman spectrum of Ni@NiO/C nanocomposite. There are two

Figure 2 (a) SEM image, (b) low- and (c) high-magnification TEM images, and (d) HRTEM image of the Ni@NiO/C nanocomposites.

characteristic peaks for the carbon-based material in Fig. 3b: the D band for “disorder” and the G band for “graphite carbon”, which are centered at 1340 and 1596 cm^{-1} , respectively. The N_2 adsorption-desorption isotherm of Ni@NiO/C nanocomposites is shown in Fig. 3c. The Barret–Joyner–Halenda (BJH) pore size distribution can be calculated from the adsorption branches (Fig. 3d). The calculated specific surface area is 301 $\text{m}^2 \text{g}^{-1}$, and the pore size distribution shows a predominant peak at 1.36 nm.

The ORR catalytic activity of Ni@NiO/C nanocomposites was investigated in 0.1 mol L^{-1} KOH solution by the three-electrode test system. The CV curves of

Figure 3 (a) XRD pattern, (b) Raman spectrum, (c) nitrogen adsorption-desorption isotherms, and (d) pore size distribution calculated from the adsorption isotherms of Ni@NiO/C nanocomposites.

Figure 4 ORR catalytic activities of Ni@NiO@C. (a) CV curves of Ni@NiO@C in N₂-saturated (black dashed line) and O₂-saturated (red solid line) 1 mol L⁻¹ KOH at a scan rate of 50 mV s⁻¹. (b) Polarization curves of Ni@NiO@C at various rotation speeds with a scan rate of 5 mV s⁻¹. (c) The corresponding KL plots at various potentials. (d) Current-time chronoamperometric response of Ni@NiO@C in O₂-saturated 0.1 mol L⁻¹ KOH at 0.3 V with a rotation speed of 1600 rpm for 15 h.

Ni@NiO@C nanocomposites were acquired in N₂-saturated and O₂-saturated 0.1 mol L⁻¹ KOH with a scan range varying from -0.1 to 1.1 V, as shown in Fig. 4a. No discernible redox peaks were found in the CV of Ni@NiO@C nanocomposites. However, the current density noticeably increased in O₂-saturated electrolyte solutions compared to that in the N₂-saturated electrolyte solutions at potentials below approximately 0.8 V. In order to investigate the kinetics of ORR, polarization curves of the Ni@NiO@C nanocomposites were surveyed at various rotation speeds with a scan rate of 5 mV s⁻¹ (shown in Fig. 4b). The limit current density increases with increasing rotation speeds (see Fig. 4b), which reveals the kinetics-limit behavior of ORR [20,21]. The diffusion layer of oxygen was thinner and more oxygen reacted on the electrode surface at high rotation speeds. The initial potential, half-wave potential, and ultimate diffusion current density are 0.87 V, 0.7 V, and 4.6 mA cm⁻², respectively, at a rotation speed of 2500 rpm. The corresponding Koutecky-Levich (KL) plots of Ni@NiO@C nanocomposites at various potentials are presented in Fig. 4c. The KL plots of Ni@NiO@C nanocomposites show strong linear relationships. The electron transfer numbers per O₂ molecule are 3.95, 3.49, and 3.97 at 0.3, 0.5, 0.7 V, respectively. These results indicate that the ORR of Ni@NiO@C nanocomposites follows a quasi-four-electron

pathway, which is similar to that of Pt/C [22]. The stability of the Ni@NiO@C nanocomposite was evaluated by monitoring the current-time chronoamperometric response in O₂-saturated 0.1 mol L⁻¹ KOH at 0.3 V with a rotation speed of 1600 rpm, which is shown in Fig. 4d. The current density can maintain 90% of its initial current density after 15 h. These results indicate that the Ni@NiO@C nanocomposite is stable and can be used in methanol and alkaline fuel cells.

The LSV measurement results for the Ni@NiO@C nanocomposite electrocatalyst are shown in Fig. 5a. The highest activity overpotential is 380 mV (vs. reversible hydrogen electrode (RHE)) under the current density of 10 mA cm⁻², which is comparable with those observed for bimetallic catalysts with excellent performances, such as Ni-Co (~390 mV) and Mn-Co (410 mV) [23]. By comparison, a similar core-shell morphological structured Co@Co₃O₄@C synthesized by Martin group has an activity overpotential of 410 mV at 10 mA cm⁻² [24]. Moreover, the current density reached 114 mA cm⁻² at a potential of 1.8 V (vs. RHE). The kinetics and mechanism of the OER are usually studied in terms of the Tafel plot. In Fig. 5b, the Tafel slope of the Ni@NiO@C catalyst is calculated to be 55 mV dec⁻¹ using a linear fit, which is much lower than those of Ni-Co (~73 mV dec⁻¹) and Mn-Co (~85 mV dec⁻¹) [23]. The result shows that the Ni@

Figure 5 Electrocatalytic OER performances of Ni@NiO@C nanocomposites: (a) OER polarization curve, (b) calculated Tafel slope, (c) cycling durability test, and (d) the chronoamperometric response measured at a current density of 10 mA cm^{-2} over 7 h.

Figure 6 Electrochemical performances of the Ni@NiO@C nanocomposites for LIB applications: (a) CV curves, (b) galvanostatic discharge/charge voltage profiles at 200 mA g^{-1} , (c) tests of rate performance at the increased rates from 100 to 1500 mA g^{-1} , and (d) cycling performance at 200 mA g^{-1} .

NiO@C nanocomposite catalyst is more efficient for the OER. Based on the LSV measurement, the CV results of the 1st and 1000th cycles are shown in Fig. 5c. After the

1000th cycle, the Ni@NiO@C catalyst shows a slight loss of current density compared to the initial LSV. The difference in the activity overpotential is just 16 mV under the

current density of 10 mA cm^{-2} , suggesting that the Ni@NiO@C architecture is highly stable over long-term OER processes [25]. Furthermore, the chronopotentiometric response curve under a current density of 10 mA cm^{-2} exhibits a steady potential at 1.59 V, as shown in Fig. 5d. The activity overpotential decreases by only 3% in a continuous experiment over 7 h. All these excellent performances of the composites indicate that although physical factors like conductivity, structural defects, and increased surface area might influence the ORR and OER activities, the C component may have a role in improving the activity of nickel [26].

Electrochemical performances of the Ni@NiO@C nanocomposites for LIB applications [25] are shown in Fig. 6. In this experiment, Li foil is used as a cathode material and Ni@NiO@C is regarded as an anode material, and the load of Ni@NiO@C is approximately 1 mg. Three consecutive CV cycles are recorded in Fig. 6a. The 2nd and 3rd cycle CV curves show similar profiles at a scan rate of 0.1 mV s^{-1} . In the first cathodic process, the initial reduction of NiO to Ni is induced by a conversion reaction ($\text{NiO} + 2\text{Li} + 2\text{e}^- \rightarrow \text{Ni} + \text{Li}_2\text{O}$) [27]. Two small peaks can be observed at 0.43 and 0.83 V, and preliminary decomposition of the electrolyte forms part of the reversible solid electrolyte interphase (SEI) layer [28]. Fig. 6b shows the galvanostatic discharge/charge performance measured at a current density of 200 mA g^{-1} between 0.01 and 3.0 V for the Ni@NiO@C nanocomposite [29]. The first discharge and charge capacities of 1125 and 683 mA h g^{-1} , are much higher than those of commercial NiO 770 and 380 mA h g^{-1} , respectively, measured at a current density of 100 mA g^{-1} [30]. To further investigate the rate capacity, the Ni@NiO@C nanocomposite electrode was tested under various current densities between 100 and 1500 mA g^{-1} , and the results are shown in Fig. 6c. The capacities of the Ni@NiO@C nanocomposite decrease as the current rates increase. Importantly, after cycling at different rates, the capacity of the Ni@NiO@C nanocomposite can recover to the initial value. Fig. 6d shows the cycling behaviors of the Ni@NiO@C nanocomposite at a rate of 200 mA g^{-1} . After 400 laps, the capacity of the Ni@NiO@C nanocomposite stabilizes at 750 mA h g^{-1} . A self-improving capacity during cycling has been found in Ni@NiO@C nanocomposites over 400 cycles, which is consistent with the observations in carbon-based anode materials [31,32]. This self-improvement ability can be attributed to the activating properties of the porous structure of the carbon layer [32]. The carbon layer can effectively decrease the charge transfer resistance and enhance the electron transfer at the interface between the

electrode and electrolyte. Therefore, the Ni@NiO@C nanocomposite presents excellent electrochemical performance as an electrode in LIBs.

CONCLUSIONS

Multifunctional carbon-encapsulated Ni@NiO nanocomposites (Ni@NiO@C) were synthesized successfully. Morphological investigations suggested that the Ni@NiO@C nanocomposites were uniform and spherically shaped core-shell structures. The current density of the Ni@NiO@C nanocomposite for ORR can maintain 90% of its initial value after 15 h in O_2 -saturated 0.1 mol L^{-1} KOH at 0.3 V with a rotation speed of 1600 rpm. From the LSV measurement, the highest activity overpotential of the Ni@NiO@C nanocomposite electrocatalyst was 380 mV (vs. RHE) under a current density of 10 mA cm^{-2} ; the current density reached 114 mA cm^{-2} at the overpotential of 1.8 V (vs. RHE); and the Tafel slope was calculated using a linear fit as 55 mV dec^{-1} . The CV results over 1000 cycles reveal that the Ni@NiO@C electrode possesses excellent electrochemical durability. Electrochemical performances of the Ni@NiO@C nanocomposite used as the LIB anode showed that the first discharge and charge capacities were 1125 and 683 mA h g^{-1} , respectively, and the Coulombic efficiency reached about 64%. After 400 laps, the capacity of the Ni@NiO@C nanocomposite stabilizes at 750 mA h g^{-1} . The conduction enhancement in OER, reduction of the pulverization effect in LIB, and improved stability in ORR of the Ni@NiO@C nanocomposite were achieved, and these advancements led to exceptional performances in OER, LIB, and ORR.

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- Conflict of interest** The authors declare that they have no conflict of interest.
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多功能碳包覆的Ni@NiO纳米复合材料用于析氧反应、氧还原反应和锂离子电池电极的研究

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摘要 本文成功合成了多功能的碳包覆Ni@NiO纳米复合材料, 并将其用作析氧反应和氧还原反应催化剂以及锂离子电池的负极材料. 扫描电镜和透射电镜照片表明Ni@NiO/C纳米复合物具有均一的球形核壳结构. 将Ni@NiO/C纳米复合物用作氧还原反应的催化剂时, 在浓度为 0.1 mol L^{-1} 的氢氧化钾溶液中以1600 rpm转速下连续反应15小时后, 电流密度仍然可以保持在初始值的90%以上. 作为析氧反应的催化剂时, 在电流密度为 10 mA cm^{-2} 时Ni@NiO/C纳米复合物的活性电位是380 mV (vs. RHE). 同时线性拟合的塔菲尔斜率为 55 mV dec^{-1} . Ni@NiO/C纳米复合物被用作锂离子电池的负极材料时, 在电流密度为 200 mA g^{-1} 下循环400圈后, 电池容量还可以达到 750 mA h g^{-1} . 结果表明, Ni@NiO/C纳米复合物用于氧还原反应、析氧反应和锂离子电池均具有优异的电化学性能.