



# Preparation of cribriform sheet-like carbon-coated zinc oxide with improved electrochemical performance



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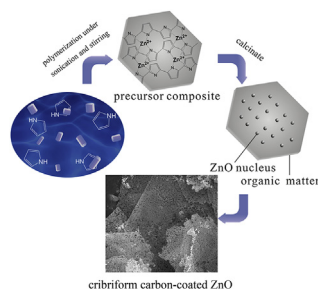
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## HIGHLIGHTS

- Carbon-coated ZnO with cribriform sheet-like morphology have been synthesized.
- As-prepared carbon-coated ZnO is evaluated as anode material for Zn/Ni rechargeable battery.
- Carbon-coated ZnO possess excellent cycle performance.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Cribriform sheet-like carbon-coated ZnO are prepared using pyrrole as the carbon source. It is found that a sheet-like precursor will form when polymerizing pyrrole in the presence of ZnO particles. After the carbonization of precursor, cribriform sheet-like carbon-coated ZnO can be obtained. Morphology and structure analysis of as-prepared carbon-coated ZnO is conducted by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The carbon overlayer not only present a barrier layer on the surface of the ZnO particles, which keeps relative high discharge capacity by inhibiting the active materials in electrode from dissolving into electrolyte, but also modify the surface status of ZnO particles so as to obtain more uniform current distribution and improved conductivity. As a result, when evaluated as an anode material for Zn/Ni cell, carbon-coated ZnO exhibit a more stable cycle performance than bare ZnO electrode.

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## 1. Introduction

The growing needs for clean energy which would be a substitute for fossil fuel has promoted the growth of innovation in the rechargeable battery field. During the last decade, the development of rechargeable batteries have grown rapidly, they can be used in numbers of applications extending from portable electronic

devices, power tools to electric vehicles. Among many different types of batteries marketed so far, not only lithium-ion batteries are attracting interest for all these uses, aqueous-based secondary batteries can also apply to a large number of nowadays applications because its unique properties. Especially, zinc electrodes are expected as a promising negative electrode since they have some features preferable as a rechargeable electrode material. For example, from the viewpoint of material resources, zinc is an abundant and low-toxic element. Furthermore, zinc has a large over-potential for hydrogen evolution reaction, which means zinc electrodes can work under the lower limit of the potential window

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of aqueous electrolytes [1,2]. As a matter of fact, zinc is the most popular negative electrode material in the field of primary battery, which also well demonstrates the virtue of zinc electrode, such as low cost, low equilibrium potential, low environmental impact and high energy density [3–6]. So zinc-based rechargeable batteries such as zinc/nickel battery are one of the most attractive electrical energy storage systems.

However, there are a few stumbling blocks in the way of utilization as rechargeable negative electrode for zinc electrode. One of the most serious problems is so called “dendrite growth” during extensive charge/discharge cycling, which results in capacity loss and even short out during circulation [7,8]. Although the electrochemistry of zinc in alkaline electrolytes is well reversible, its non-uniform dissolution and deposition usually results in dendritic growth [9–11]. Many different approaches have been attempted in order to avoid the dendrite growth. Modifications to the electrode or electrolyte have been carried out so as to better retain the discharge product rather than dissolve into electrolyte. Addition of metal oxides and hydroxides to zinc electrode, such as  $\text{Ca}(\text{OH})_2$ ,  $\text{Bi}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{Ag}$ ,  $\text{In}_2\text{O}_3$ ,  $\text{HgO}$ ,  $\text{PbO}$ ,  $\text{CdO}$  and  $\text{In}(\text{OH})_3$ , has long been recognized as effective [12–18]. These additives are insoluble in alkaline electrolytes, and therefore retain a uniform distribution during the battery cycle. Some of them will form insoluble compound with the zincate ions, so that maintaining zinc species in the proximity of zinc electrode. Some of them could be reduced and formed an electronic network before zinc deposition, which could in turn enhance the electronic conductivity and decrease polarization of the electrode. Besides the metal oxides or hydroxides, organic additives to the electrode or electrolyte have been investigated for enhancing cycle performance of zinc electrode [19–22]. Ghavami and co-workers studies the effects of different kinds of surfactant and derivatives of benzene on the electrochemical properties of electrolytic zinc, they suggested that the positive head group of cationic surfactants promotes the cathodic current distribution, which may prevent dendritic growth [23]. Vatsalarani and co-workers reported the dip coating, brush coating and electrodeposition coating of polyaniline or polypyrrole on a porous zinc electrode which allowed the movement of hydroxide ions but restricted the diffusion of zincate ions [24,25].

From above researches, we perceive that the dendrite growth zinc electrode is tightly related to the surface states of ZnO particle, it is reasonable that the dissolution and electro-deposition process are believed to firstly occur at surfaces of ZnO particles. Carbon coating is useful surface modification techniques for anode and cathode materials in lithium ion batteries because that the carbon coating layers can significantly improve the electronic conductivity of active materials, and result in improved electrochemical performance of electrode [26,27]. But there is few researches reporting the carbon-coated material for Zn/Ni battery. In addition, carbon material is insoluble in alkaline electrolyte, it is also helpful to be used as anode material for alkaline battery. So, in the present study, the carbon-coating ZnO with novel cellular sheet-like morphology was prepared using pyrrole as carbon source. The electrochemical performances of Zn/Ni cell with the as-prepared carbon-coated ZnO electrode were investigated.

## 2. Experimental section

### 2.1. Preparation of cribriform sheet-like carbon-coated ZnO

In order to obtain the uniform carbon coating over the surface of ZnO particles, pyrrole and ZnO were dispersed with continuous sonication and mechanical stir, then oxidant was added to polymerize the pyrrole. Afterward, the obtained precursor suffers from carbonization to obtain final product. In a typical synthesis, a

certain amount of ZnO (Xilong chemical Co., Ltd.) was dispersed in 20 mL distilled water under ultrasound, 3.7, 7.4 and 11.1 mmol pyrrole monomer (Sinopharm Chemical Reagent Co., Ltd.) were respectively added to above ZnO suspended solution under constant sonication and continuously stirred for 5 min. The product prepared with different initial pyrrole content is coded as Sample 1 (3.7 mmol pyrrole), Sample 2 (7.4 mmol) and Sample 3 (11.1 mmol), respectively. Afterward, 1.8, 3.6, and 5.4 mmol ammonium persulfate (APS, Sinopharm Chemical Reagent Co., Ltd.) was promptly mixed into the above solution at room temperature. The resulting solution was kept under ultrasound and stirred for 30 min. During the polymerization process, the color of the system changed from white to black gradually. The samples were filtered and washed thoroughly with distilled water and methanol (to remove the unreacted ammonium persulfate and sodium p-toluene sulfonate). After the product was vacuum-dried at 333 K temperature, black powder was obtained. For carbonization, a certain amount of as-obtained powder was loaded into a tube furnace and kept at 973 K for 8 h under  $\text{N}_2$  atmosphere, then the final products were obtained. The carbon content of samples is determined by the difference in weight before and after calcinations treatment in the oxygen atmosphere at 873 K for 90 min, it is 3.4%, 9.7% and 19.2% for Sample 1, Sample 2 and Sample 3, respectively.

The ZnO electrodes were prepared by pasting the slurries (as-prepared carbon-coated ZnO, acetylene black and PTFE) at a copper mesh substrate. The weight ratio of active material, acetylene black and PTFE was 85:10:5. The obtained electrodes were dried at 333 K in a vacuum oven for further testing.

### 2.2. Characterization and electrochemical measurements

The morphology of the samples was observed using a field emission scanning electron microscope (FESEM) (JSM-6360LV) and a transmission electron microscope (TEM) (JEOL-2010). The X-ray diffraction was carried out with a X-ray diffraction (Philips) using  $\text{Cu K}\alpha$  radiation at 35 kV and 200 mA,  $\lambda = 1.5406 \text{ \AA}$ . The galvanostatic charge/discharge cycle test was conducted on a battery test apparatus NEWARE BTS-610 (Neware Technology Co., Ltd., China), the cells were charged at 1C for 60 min and discharged at 1C down to 1.2 V cut-off (or charged at 3C for 20 min and discharged at 3C down to 1.2 V cut-off). The cyclic voltammetry test (CV) was carried out using RST 5000 (Zhengzhou Shiruisi Technology Co., Ltd.) electrochemical workstation over the range from  $-0.85 \text{ V}$  to  $-1.65 \text{ V}$  with scanning rate of  $20 \text{ mV s}^{-1}$ . Electrochemical impedance spectroscopy (EIS) was conducted with Parstat 2273 (Princeton Applied Research) electrochemical workstation over the frequency range from 100 kHz to 0.1 Hz, the amplitude of AC potential perturbation was 10 mV. The EIS data was fitted using the software of Zview. In the electrochemical measurements, as-prepared ZnO electrodes serves as the working electrode, a sintered  $\text{Ni}(\text{OH})_2$  electrode as counter electrode, 6.0 M KOH solution with saturated ZnO was used as the electrolyte, multilayer polypropylene microporous membranes as the separator and an Hg/HgO electrode as reference electrode.

## 3. Results and discussions

### 3.1. Material characterizations

The diffraction patterns for bare ZnO and carbon-coated ZnO are shown in Fig. 1. It can be seen that there is a group of characteristic diffraction peaks for ZnO in the all XRD spectra of carbon-coated ZnO, which is well consistent with the diffraction peaks of bare ZnO sample (JCPDS card 36-1451 hexagonal wurtzite structure), indicating that carbon-coated ZnO remains the fine crystallinity of

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