FULL PAPER

Synthesis and electrochemical properties of nickel oxide coated ZnMn_2O_4 nanocomposites

Yongjie LI^{1,2}, Zhiqiang WEI^{1,2,†}, Meijie DING², Long MA², Xueliang ZHU² and Jiahao LIANG²

¹State Key Laboratory of Advanced Processing and Recycling of Non-ferrous Metals, Lanzhou University of Technology, Lanzhou 730050, China

²School of Science, Lanzhou University of Technology, Lanzhou 730050, China

Nickel oxide coated ZnMn_2O_4 nanocomposites were successfully synthesized by two-step method combined hydrothermal reaction and calcination process. The microstructure, morphology and the color mapping of the as-prepared samples were characterized by means of X-ray diffraction, high resolution electronic microscope microstructure, field emission scanning electron microscopy and energy-dispersive X-ray spectrum. The electrochemical performance of the samples were investigated by cyclic volt-ampere (CV), constant-current charge-discharge and electrochemical impedance spectroscopy. The experimental results show that nickel oxide coated ZnMn_2O_4 nanocomposites exhibit weak Ni_2O_3 hexagonal phase besides obvious ZnMn_2O_4 spinel tetragonal structure, the morphologies are uniform polyhedron structure with obvious cladding layer. CV curves exhibit rectangular shape without obvious redox peaks. Nickel oxide coated ZnMn_2O_4 nanocomposites shows better specific capacitance and excellent cyclic stability, and maintains 98.8% of its initial capacitance after 1200 cycles. Nyquist plots of nickel oxide coated ZnMn_2O_4 nanocomposites proving the samples possess lower resistance and ideal electron conductivity.

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1. Introduction

In recent decades, with the rapid growth of global population and economy, the energy crisis and environmental pollution have become increasingly serious, and the development and utilization of efficient and clean new energy sources have received increasing attention.^{1)–5)} Supercapacitors is a new type of energy storage devices between electrolytic capacitors and rechargeable batteries,^{6)–8)} which has attracted extensive attention as unique electrochemical energy storage devices due to its long cycling life, large power density, moderate energy density, fast charge-discharge speed and wide operating temperature range.^{9)–14)} Hence, several materials have been explored as electrode materials for energy storage applications. In contrast, spinel mixed metal oxides have been extensively studied and applied as electrode materials for supercapacitors due to their higher specific capacity than carbon materials and better cyclic stability than conductive polymers.^{15)–20)}

ZnMn_2O_4 is a typical spinel type mixed metal oxide with narrow band gap of 1.86 eV, and belonging to I41/amd space group.²¹⁾ It was demonstrated that ZnMn_2O_4

nanocrystals exhibit higher specific capacitance, but the cycle stability is poor,^{22),23)} while transition metal oxide possess better cycle stability, but the specific capacitance is lower.^{24)–26)} Therefore, it is very significative to combine the advantages of the two types of spinel type mixed metal oxide and transition metal oxides to make them have higher specific capacitance and better cycle stability.^{27)–29)} In particular, nanostructures have great potential to achieve excellent electrochemical properties due to nanostructures can provide relatively short diffusion paths and relatively high surface area.^{30),31)} Unfortunately, to the best of our knowledge, there are few reports on the electrochemical properties of nanocomposites about spinel type mixed metal oxides and transition metal oxides.

In this paper, nickel oxide coated ZnMn_2O_4 nanocomposites with spinel tetragonal structure were successfully synthesized by two-step method combined hydrothermal reaction and calcination process. The samples were evaluated by means of X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and energy-dispersive X-ray spectrum, high resolution electronic microscope microstructure (HRTEM), cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), cycle stability and electrochemical impedance spectra (EIS) to compare the microstructure and supercapacitor perform-

[†] Corresponding author: Z. Wei; E-mail: 2667355047@qq.com

ances of ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ nanocomposites.

2. Experimental

2.1 Materials preparation

Nickel oxide coated ZnMn₂O₄ nanocomposites were synthesized by two-step method combined hydrothermal reaction and calcination process. In a typical procedure, according to the molar ratio of the metal cations (the atomic weight ratio of Ni:Mn in this experiment is 5%), weighed stoichiometric amounts of Zn(NO₃)₂·6H₂O, Ni(NO₃)₂·9H₂O and Mn(NO₃)₂·6H₂O used as precursors. ZnMn₂O₄ nanocrystals were synthesized via a facile hydrothermal method as described in literature.²²⁾ A certain amount of nickel nitrate was weighed and dissolved in 100 mL anhydrous ethanol under constant magnetic stirring for 30 min. Then slowly added ZnMn₂O₄ powders to the above solution, and the suspension solution was evenly dispersed by magnetic stirring at room temperature for 1 h. Subsequently, the resultant solution was transferred to and sealed in a Teflon-lined stainless-steel autoclave of 100 mL capacity and submitted to hydrothermal treatment at 100°C. After reaction for 12 h, the autoclave was naturally cooled down to room temperature. Thereafter, the produced precipitate was separated centrifugally and washed several times with distilled water and absolute alcohol, respectively, and then dried at 60°C in a vacuum oven for 10 h. Finally, the products were heated treatment at 700°C for 6 h in the tube furnace. Thus, nickel oxide coated ZnMn₂O₄ nanocrystals were collected and used for further studies.

2.2 Characterization

The crystallographic data for the products were examined by XRD with CuK α radiation at $\lambda = 0.154056$ nm. The 2θ range scanned was 20 to 80° with scanning rate 0.005°·s⁻¹ and step size 0.02°. The morphology and the atomic percentage of the compositions along with the color mapping of the samples was investigated by HRTEM (JEOL JEM-2010), FESEM (JEOL JSM-6701F) and Energy dispersive analysis of X-ray.

The electrochemical performance of were tested on an electrochemical workstation (CS350, Wuhan CorrTest, China) in Na₂SO₄ aqueous solution with a concentration of 1 M as the electrolyte. Three-electrode cell configuration which including working electrode, counterelectrode, and reference electrode. Platinum foil served as the counter electrode, and standard calomel electrode served as the reference electrode, respectively. The work electrode was fabricated as follows: active substance, acetylene black and polyvinylidene fluoride were mixed at weight ratio of 8:1:1 using ethyl alcohol as solvent to form slurry. The slurry was uniformly coated on the surface of nickel foam. Then the meshes were dried at 80°C for 12 h and pressed under 10 MPa. The mass of the samples on the Ni foam electrode was 2 mg·cm⁻² as the working electrode. The electrochemical performance of the as-prepared samples was investigated by CV, GCD and EIS.

3. Results and discussion

Figure 1 shows the XRD patterns of bare ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ nanocomposites, as well as the standard XRD patterns of ZnMn₂O₄ tetragonal structure (PDF #71-2499) and Ni₂O₃ hexagonal structure (PDF #14-0481). For bare ZnMn₂O₄ sample, all the diffraction peaks can well correspond to the standard diffraction pattern of tetragonal ZnMn₂O₄ (space group: I4₁/amd, $a = b = 0.5720$ nm, $c = 0.9245$ nm). There is no impurity diffraction peak in the pattern, indicating a high purity of the sample. It can be found that ZnMn₂O₄ nanocrystals before and after coating nickel oxide coating did not cause structural change, and maintain the tetragonal structure. In addition, additional weak diffraction peaks from Ni₂O₃ hexagonal phase can be seen in the XRD pattern of the composite, which indicates that Ni₂O₃ nanoparticles are formed in the composite.

The SEM images of ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ nanocomposites are shown in **Fig. 2**. It can be seen that bare ZnMn₂O₄ exhibits irregular polyhedron shape with average size of several hundred nm. The surface is rough and unclear with obvious agglomeration phenomenon. A large number of particles densely piled together and forming honeycomb-like morphology. **Figure 2(b)** presents SEM image of nickel oxide coated ZnMn₂O₄ nanocomposites, revealing that the particles present uniform polyhedron structure with clear boundaries and smooth surface, the microstructure and morphology of ZnMn₂O₄ sample after coating treatment without change, but the particle boundary become clear with a certain degree of agglomeration.

Figure 3(a) shows the dark-field FESEM image of nickel oxide coated ZnMn₂O₄ nanocomposites, **Figs. 3(b)–3(e)** give the color mapping image of corresponding elements in the area indicated in **Fig. 3(a)**. Morphology of nickel oxide coated ZnMn₂O₄ composites reveals regular polyhedron structure with more or less agglomeration. The element mapping images of the composite material

Fig. 1. XRD patterns of ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ nanocomposites.

Fig. 2. SEM images of (a) ZnMn_2O_4 and (b) nickel oxide coated ZnMn_2O_4 nanocomposite.

Fig. 3. (a) FESEM image and (b–e) the corresponding elemental mapping images of nickel oxide coated ZnMn_2O_4 nanocomposites.

confirms that Zn, Mn, O and Ni elements are evenly distributed, and the composite material without obvious chemical composition segregation in the composite phase according to the molar concentration of its precursor. In addition, the uniform distribution of Ni elements on the whole micro-cuboid further proves that nickel oxide has a uniform coating on the surface of ZnMn_2O_4 .

Figure 4 shows the HRTEM observation results of nickel oxide coated ZnMn_2O_4 composites. It can be seen from Fig. 4(a) that nickel oxide coated ZnMn_2O_4 nanoparticles are closely aligned together with clear edges and smooth surface, most of the nanoparticle surface have obvious surface coating layer. Figure 4(b) displays the local high magnification HRTEM micrographs of nickel oxide coated ZnMn_2O_4 composites. It is evident that Ni_2O_3 encapsulated ZnMn_2O_4 nanoparticles with obvious cladding structure. For Ni_2O_3 and ZnMn_2O_4 nanoparticles, the perfect lattice fringes are clearly visible, which implying that the sample has good crystallization without obvious internal defects. The interlayer distance is about 0.25 nm, which is well matched with the inter planar distance of the (211) lattice planes of ZnMn_2O_4 tetragonal structure, and

exhibit a well oriented growth along the (211) direction, while the thickness of cladding layer approximately 6 nm, and the lattice spacing of the crystalline cladding layer is about 0.28 nm, which is well agreement with values the (002) lattice planes of Ni_2O_3 hexagonal structure. HRTEM observation results are consistent with the XRD results, which further indicating that ZnMn_2O_4 nanocrystals were successfully coated with nickel oxide.

Figure 5 shows the CV curves of ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 electrodes at various scan rates ranging from 5 to $100 \text{ mV} \cdot \text{s}^{-1}$ in the voltage window of -0.1 – 0.7 V . It is obviously that the CV curves of ZnMn_2O_4 samples before and after coating present similar symmetric rectangular shapes without any redox peaks at all scan rates, the CV curves mean ZnMn_2O_4 electrode exhibit a rapid and reversible reaction, easy ion transport and fast spread because of its better electrical double-layer capacitance feature, and showing excellent electrochemical reversibility and ideal capacitive behavior.

Figure 5(c) shows the plots of the specific capacitance values for ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 composites in proportion to CV profile. It is easily found

Fig. 4. HRTEM images of nickel oxide coated ZnMn_2O_4 composites.

Fig. 5. CV curves of (a) ZnMn_2O_4 , (b) nickel oxide coated ZnMn_2O_4 , (c) specific capacitance of samples at different scan rates, (d) CV curves of ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 nanocomposites.

that the specific capacitance of ZnMn_2O_4 electrodes decreases gradually with the increase of the scan rate. On the one hand, the energy storage efficiency of the electrode is low due to the insufficient ion transfer between the electrode and the electrolyte interface, on the other hand, the charge transfer reduces the diffusion rate of electrolyte into each electrode materials at higher scan rate. Moreover, the specific capacitance of nickel oxide coated ZnMn_2O_4 is obviously better than that of pure ZnMn_2O_4 at given scan rate. When the scanning rate is $5 \text{ mV} \cdot \text{s}^{-1}$, the estimated maximum specific capacitance for ZnMn_2O_4 and nickel

oxide coated ZnMn_2O_4 samples are 133 and $193 \text{ F} \cdot \text{g}^{-1}$, respectively. Meanwhile, with the scanning rate increasing from 5 to $200 \text{ mV} \cdot \text{s}^{-1}$, the capacitance retention rates of ZnMn_2O_4 and nickel oxide coating ZnMn_2O_4 samples maintain 55 and 75% , respectively. In addition, the specific capacitance of nickel oxide coated ZnMn_2O_4 is obviously better than that of ZnMn_2O_4 electrodes at a certain scanning rate. Compared with ZnMn_2O_4 samples, the superior specific capacitance of nickel oxide coated ZnMn_2O_4 composites mainly due to the regular polyhedron architecture with small particle size and large specific surface

Fig. 6. GCD curves of (a) ZnMn₂O₄ and (b) nickel oxide coated ZnMn₂O₄ samples at various current densities. (c) GCD curves of ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ composites at the current densities of 2.5 A·g⁻¹, (d) specific capacitance of samples at various current densities.

area, which provides more active sites for Faradaic reactions, improve the movement of ions and electrons, thus promoting the electrochemical properties.

In order to further study the advantages of ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ composites, the CV curves for ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ composites were recorded at scan rate 5 mV·s⁻¹ as shown in Fig. 5(d). Obviously, the specific capacitance is proportional to the average area of a CV curve, and the CV loop area of nickel oxide coated ZnMn₂O₄ composites is much larger than that of ZnMn₂O₄, which corresponding to the excellent specific capacitance than pure ZnMn₂O₄ samples.

It can be seen that nickel oxide coating can not only change the structure of ZnMn₂O₄, but also improve the surface and pore structure of the material. Meanwhile, the existence of nickel oxide coating increases the penetration capacity of cationic ions from the electrolyte to electrode material, and greatly improve the chemical reaction of the electrolyte ions and the surface of the electrode material, which can provide a better electrolyte ion channel, so its energy storage capacity is also better, which can provide a better electrolyte ion channel, and make it have better energy storage capacity.

Figure 6 present the GCD curves for ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ samples obtained at various

current densities ranging from 0.25 to 3.5 A·g⁻¹ over the potential window between 0 and 0.48 V at different current densities. It is clearly that the curves for ZnMn₂O₄ samples before and after coating resemble each other quite closely with a nearly linear and isosceles triangle, the charge curves and discharge curves are symmetric at all current densities. The symmetry of the curves indicates that the samples have good invertibility, and the linear nature of the curves suggests that the electrochemical reaction are caused by the super capacitance behavior, which is concordant with the CV results shown in Fig. 4. The GCD curves for ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ samples at current density of 2.5 A·g⁻¹ are illustrated in Fig. 6(b), it can be seen that charge and discharge time of nickel oxide coated ZnMn₂O₄ is longer and the charge and discharge efficiency is higher than that of ZnMn₂O₄.

Figure 6(d) shows the specific capacitance of ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ at various current densities. The specific capacitance of nickel oxide coated ZnMn₂O₄ electrode is obviously higher than that of ZnMn₂O₄ at given current density. When the current density 0.25 A·g⁻¹, the specific capacitance of ZnMn₂O₄ and nickel oxide coated ZnMn₂O₄ samples are about 186 and 100 F·g⁻¹, respectively. With the increase of current density, the specific capacitance exhibited a decreasing trend, when the current density increases to 3.5 A·g⁻¹, the

Fig. 7. Cycle stability of ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 nanocomposites at current density of $2.5 \text{ A} \cdot \text{g}^{-1}$ over 1200 cycles.

specific capacitance of the samples decreases to 110 and $90 \text{ F} \cdot \text{g}^{-1}$, respectively. In addition, the specific capacitance of nickel oxide coated ZnMn_2O_4 electrode at given current density is significantly higher than that of ZnMn_2O_4 electrode, nickel oxide coated ZnMn_2O_4 electrode exhibit excellent electrochemical performance at high charge–discharge current density. This can be explained by the relatively small size and good dispersion of the regular polyhedron structure, which can provide more available electrochemical reaction of the active site, and lead to the increase of ionic resistivity and reduce charge diffusion of the inner active site and redox reaction rate.

The electrochemical cycling stability for ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 as electrode material were evaluated by the repeated charge–discharge measurement at constant current density of $2.5 \text{ A} \cdot \text{g}^{-1}$ for 1200 cycles. **Figure 7** exhibits the capacitance retention as a function of the charge–discharge cycling number. The initial specific capacitance for ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 samples is 96 and $167 \text{ F} \cdot \text{g}^{-1}$, respectively. After 1200 cycles, the specific capacitances of the samples still remain about 90 and $165 \text{ F} \cdot \text{g}^{-1}$, and maintain about 94 and 99% of its initial value, respectively. Compared with pure ZnMn_2O_4 , nickel oxide coated ZnMn_2O_4 nanocomposites exhibit better electrochemical performance and stability.

Nickel oxide coated ZnMn_2O_4 nanocomposites exhibit excellent electrochemical performance and stability compared with pure ZnMn_2O_4 owing to the synergistic effect. On one hand, nickel oxide as active sites coated the surface of ZnMn_2O_4 particles and the integration of nickel oxide with ZnMn_2O_4 can supply more active sites due to the coexistence of tetrahedral sites and octahedral sites, which can facilitate rapid electrochemical reaction of active material and improve rapid electronic transport from electrode material to the current collector. On the other hand, the regular polyhedron architecture with small particle size and large specific surface area can improve electrochemical behavior and provide more pathways for charge transfer in electrolyte.

Fig. 8. Nyquist plots of ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 nanocomposites.

Figure 8 shows Nyquist plots for ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 samples measured in frequency range from 0.01 Hz to 100 kHz , the inset shows the enlarged high frequency region of the spectra and the equivalent circuit. The equivalent circuit including the solution resistance (R_s), charge-transfer resistance (R_{ct}), double layer capacitance (C_d), pseudo capacitance (C_p) and Warburg impedance (W). The Nyquist plots of the impedance spectra contains a semicircle at the high frequency region and a straight line at the low frequency region, where the diameter of the semicircle represents the charge-transfer resistance at the electrode–electrolyte interface, and the slope of the straight line at low frequency represents the Warburg impedance of electrolyte ions diffused into the electrode. The diameters of the semicircle for ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 samples are very small (inset of Fig. 7), indicating that the R_{ct} value is very small, these results indicate that the electrode possess better electrical conductivity, and the Faradic reaction is easy to carry out and has a high reversibility. The phase angle of Nyquist plots for all samples in the mid frequency region higher than 45° as illustrated from the inset of Fig. 8, which indicates an important capacitive behavior of the sample. In addition, the intersection of Nyquist plots at the x -axis represents the internal resistance (R_s), including the ionic resistance of the electrolyte, the intrinsic resistance of electrode materials and the contact resistance at the electrode–electrolyte interface. The R_s values of ZnMn_2O_4 and nickel oxide coated ZnMn_2O_4 samples is approximately 3.11 and 3.39Ω , respectively. The R_s value for nickel oxide coated ZnMn_2O_4 samples is the lowest, indicating that the sample exhibit rapid electron transfer and the best electrical conductivity, as well as excellent specific capacitance.

4. Conclusions

Nickel oxide coated ZnMn_2O_4 nanocomposites were successfully synthesized by two-step method combined hydrothermal reaction and calcination process. Nickel

oxide coated ZnMn_2O_4 nanocomposites exhibit weak Ni_2O_3 hexagonal phase besides obvious ZnMn_2O_4 spinel tetragonal structure, the morphologies are uniform polyhedron shape with obvious cladding layer, and the thickness of cladding layer about 6 nm. The CV curves are similar to rectangular shape without significant redox peaks. Nickel oxide coated ZnMn_2O_4 electrode exhibits better specific capacitance and excellent cyclic stability, and maintains 98.8% of its initial capacitance after 1200 cycles. Nyquist plots of the samples indicating nickel oxide coated ZnMn_2O_4 nanocomposites exhibit lower resistance and ideal electron conductivity.

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