Amorphous Cobalt Boron Alloy@Graphene Oxide Nanocomposites for Pseudocapacitor Applications

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Amorphous Co–B alloy nanoparticles grown on graphene sheets were synthesized via a chemical reduction approach and successfully used for an application as a pseudocapacitor. This study aims to improve the capacity and cycling stability of amorphous Co–B alloy nanoparticles grown on conductive graphene sheets. The products were characterized by X-ray powder diffraction, scanning electron microscopy and transmission electron microscopy. As electrode materials for pseudocapacitors, the amorphous Co–B alloy grown on graphene oxide (Co–B@GO) exhibits a high specific capacitance of 460 F g^{-1} , which is nearly 1.5 times greater than that of bare Co–B nanoparticles at 1 A g^{-1} . The specific capacitance preserved 84% of the initial capacitance even after 1000 cycles at a scan rate of 10 m V^{-1} , suggesting its promising potential as pseudocapacitor materials.

Key words: Pseudocapacitor, Electrode, Cobalt–boron alloy, Amorphous alloy, Graphene oxide

1. Introduction

Electrochemical capacitors (so-called supercapacitors) have become one of the most promising candidates for next-generation power devices, with desirable properties of high power density, fast

charging rate and excellent cycling stability^[1,2]. Based on the charge storage mechanism, supercapacitors are classified as electrochemical double layer capacitors (EDLCs) with physical adsorption of ions at the interface of the electrode surface and the electrolyte, and pseudocapacitors with a fast reversible faradaic charge transfer at the electrode surface^[3]. Currently, most commercial supercapacitors are EDLCs that are based on carbon materials^[4,5]. However, the low energy density of these devices has so far barred them from many important applications^[6]. Transition metal oxides such as MnO_2 ^[7], CuO ^[8], ZrO_2 ^[9], and SnO_2 ^[10], are a group of very promising pseudocapacitor electrode materials to replace expensive and toxic RuO_2 . Although they exhibit higher specific capacitance and energy density than EDLCs, they usually suffer from poor cyclability due to irreversible redox reactions at the surface of active materials^[11].

A recently adopted approach to overcome these issues is to develop composites of carbon-based materials and active materials as hybrid electrodes, which combines advantages of EDLCs and pseudocapacitors^[8,12]. In the carbon materials, graphene is an ideal matrix with many fascinating properties, such as extraordinarily high electrical conductivity, large specific surface area, high mechanical flexibility, and pronounced thermal and chemical stability^[13,14]. Therefore, much research has focused on the fabrication of composites based on graphene for pseudocapacitor applications, generating special properties by the reinforcement or modification of each other. For example, Sahu et al.^[15] reported the growth of interconnected ZnO nanorings initiated through functionalities present in the edge planes of lacey graphene nanoribbons (GNRs). The test results show that a ZnO/GNR nanocomposite electrode exhibits a 2.0 V potential window in an aqueous electrolyte and an ultra-short time constant (0.08 s) in an asymmetric supercapacitor device with lacey reduced graphene oxide nanoribbons (LRGONRs) as a negative electrode. Furthermore, the ZnO/GNR nanocomposite electrode exhibits high capacitance and cycling stability as compared with ZnO synthesized by the same procedure without the support LGONR.

Compounds of cobalt exhibit exceptional physio-chemical properties and have been widely applied in fabricating pseudocapacitor electrode^[16,17]. For instances, Mai et al.^[18] synthesized the three-dimensional (3D) multicomponent oxide, $\text{MnMoO}_4/\text{CoMoO}_4$. They fabricated asymmetric supercapacitors based on hierarchical $\text{MnMoO}_4/\text{CoMoO}_4$ heterostructured nanowires, which show a specific capacitance of 187.1 F g^{-1} at a current density of 1 A g^{-1} , and good reversibility with a cycling efficiency of 98% after 1000 cycles. Tian et al.^[19] established a simple and efficient strategy

to achieve both ultrahigh energy and power densities of micropseudocapacitors (MPCs) with the plantation of pillar arrays in the microspirals with ultrathin nanosheets. A compound of cobalt as the microelectrode delivers both ultrahigh energy density of about 34.9 mWh cm^{-3} at a scan rate of 10 mV s^{-1} and high power density of 193.4 W cm^{-3} at the ultrahigh scan rate of 200 V s^{-1} . In addition, it also demonstrates an excellent cycling stability. However, researchers seldom attempt to fabricate Co_xB_y as electrode materials for pseudocapacitor application. Liu et al.^[20] reported that a series of Co–B alloys were synthesized successfully by arc melting, resulting in good cycling stability. However, this approach is not suitable for mass production due to their high synthesis costs as well as present technological difficulties. Wang et al.^[21] synthesized ultrafine amorphous alloy powders of Co–B and tested them for possible application as electroactive anodic materials. The results demonstrated that the Co–B particles exhibit excellent electrochemical reversibility and a high charge–discharge capacity. Moreover, we^[22] reported that mesoporous carbon electrodes can be doped with Co–B nanoparticles via a chemical reduction approach and successfully used for pseudocapacitor application, which presents a maximum specific capacitance of 531 F g^{-1} at a scanning rate of 5 mV s^{-1} . However, the cycling performance of amorphous Co–B alloy has limited its commercial attractiveness for pseudocapacitor applications.

In this study, we prepared nanocomposites of Co–B particles grown on graphene support, based on an oxidation–reduction reaction between graphene oxide and CoCl_2 . The electrochemical properties of the Co–B@GO nanocomposites as pseudocapacitor electrodes were investigated by a series of electrochemical tests.

2. Experimental

2.1 Synthesis of graphene oxide

Graphene oxide (GO) was synthesized according to the Hummers method^[23]. The resulting slurry was centrifuged and washed with 2 mol/L HCl until it was SO_4^{2-} free. The acid-washed GO was re-dispersed in water to form a brown dispersion. The pH of the dispersion was then adjusted to pH of 5–6 with concentrated ammonia solution to promote exfoliation of GO, and unoxidized graphite impurities were removed by centrifugation at 4000 rpm. The pH of the resulting supernatant

was adjusted back to pH of 3, and the supernatant was dialyzed several times in deionized (DI) water until its pH became close to 5^[24]. The dispersion was then diluted by methanol to obtain a GO concentration of 1 mg/mL.

2.2. *Synthesis of amorphous Co–B alloy @GO*

All chemicals were of analytical grade, and were used without any further purification. The typical synthesis procedure of amorphous Co–B alloy@GO nanocomposite is similar to our previous work^[22]. CoCl₂ 6H₂O (1.0 g) was dissolved in distilled water (4 mL). Then, 0.5 g of GO was added to the resulting solution, dipped for 12 h (denoted as compound 1). Ethanol solution (50 mL) of sodium borohydride (0.1 mol/L) was first prepared and adjusted to pH of 12 with sodium hydroxide to prevent violent hydrolysis. Compound 1 (0.8675 g) was dissolved in 5 mL of ethanol solution and then cooled in an ice bath. The chemical reduction reaction was carried out by adding borohydride solution dropwise into the metal salt solution while being stirred. When the addition was complete, the solution bath was continually stirred for about 1 h to under N₂ flow to release the hydrogen. The resulting solution was centrifuged, and the precipitate was washed three times with distilled water, followed by three washing steps with ethanol. Finally, the sample was stored in absolute ethanol before use (denoted as Co–B@GO).

2.3. *Electrochemical measurements*

Pseudocapacitor electrodes were constructed by mixing the active material, carbon black and poly tetrafluoroethylene (PTFE) binder with a weight percent ratio of 75:20:5, and then the mixture was dispersed in alcohol and was pressed onto nickel foam under a pressure of 12 MPa. The geometric surface area of the prepared working electrode was 1 cm². The electrodes were dried under vacuum at 90 °C for 1 h to remove the solvent. Nickel foam (1.6 mm thick, 95% purity, Goodfellow) was used as a current collector.

Cyclic voltammetry (CV), galvanostatic charge/discharge and electrochemical impedance spectroscopy (EIS) measurements were carried out with a CHI660D electrochemical workstation (ShanghaiChen Hua, Inc.). A conventional three-electrode cell was equipped with a Pt plate as the

counter electrode and a saturated calomel electrode (SCE) electrode as the reference electrode. All experiments were carried out at room temperature and in 6 mol/L KOH solution.

2.4. Compositional and structural characterization

Powder X-ray diffraction (PXRD) of the samples was performed on a D/Max-RB diffractometer with CuK_α radiation ($\lambda=0.154056$ nm) and a graphite monochromator at 50 kV, 100 mA. The size and morphology of the powders were observed by transmission electron microscope (TEM) on a Hitachi H-800 microscope and scanning electron microscopy (SEM) on a JEOL-6700 microscope operating at 5 kV.

3. Results and Discussion

3.1. Material characterization

X-ray diffraction (XRD) was employed to determine the structures of the studied samples. Fig. 1 shows that the diffraction peaks of GO appear at around 2θ of 10° and 42° , corresponding to the (002) and (111) reflections of GO, respectively, which is in accord with previous reports^[25,26]. (002) and (100) diffraction peaks in graphene indicate that the synthesized graphene did not perfectly exist as a single layer^[26]. It can be seen that the XRD pattern of Co-B@GO does not exhibit distinct peaks, which is consistent with our previous report^[22]. According to a report by Shen and Chen^[27], the results show that the average particle size of as-prepared CO-B and Co-B@GO is approximately 4 nm. Moreover, no conventional stacking peaks of GO at $2\theta=10^\circ$ and 42° were detected in Co-B@GO, suggesting that the graphene sheets were homogeneously dispersed onto the surface of the Co-B nanoparticles^[28].

The detailed crystal structures were further examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). In the SEM image of GO (Fig. 2(a)), a crumpled layered structure with several stacking layers is observed. The TEM image (Fig. 2(b)) shows that the Co-B nanoparticles are spherical particles with primary diameters between 3 and 5 nm. This result is in agreement with the XRD results (Fig. 1) and a previous report^[22]. The amorphous characteristics

of the Co–B can be further confirmed by selected-area electron diffraction (SAED), which shows no dots or rings, indicating amorphous components. In addition, it clearly shows that the nanoparticles aggregated to a large extent, which is detrimental to the electrochemical properties. Fig. 2(c, d) show the TEM images of the as-prepared CO–B@GO nanocomposite. As estimated from the TEM image, the average size of the Co–B@GO nanostructures is similar to that of Co–B. However, it also can be seen from the images (Fig. 2(c, d)) that aggregation of the Co–B nanoparticles is slight. The Co–B nanoparticles are highly dispersed on the GO and their dispersion is much better than the bare amorphous Co–B alloy. The inset in Fig. 2(c, d) shows the SAED patterns of GO and the Co–B@GO nanocomposite, respectively. To compare the SAED test results, no other diffraction points was observed in the SAED of Co–B@GO, indicating that amorphous Co–B alloy is loaded on the GO. The well-dispersed nanoparticles of amorphous Co–B alloy on the highly conductive GO will enhance both the ionic diffusion and electrical conduction and improve their electrochemical performance.

3.2. Electrochemical characterization

The prepared samples were evaluated as the active material in pseudocapacitors by employing cyclic voltammetry (CV) measurements. Fig. 3(a) shows the CV of prepared Co–B and Co–B@GO electrodes, recorded in 6 mol/L KOH electrolyte at a scan rate of 5 mV s⁻¹. It is clearly seen that the Co–B@GO nanocomposites exhibit much higher current intensity than Co–B, suggesting the Co–B@GO exhibits higher specific capacitance. Fig. 3(b) shows the CV curves of the Co–B@GO composite electrode (the absolute weight of the composites is 1.176 mg) measured at different scan rates of 5, 10, 20, 30, 40 and 50 mV s⁻¹ between -0.2 and 0.6 V. Then, owing to the appearance of the redox peak pair, it reveals pronounced pseudocapacitive characteristics different from the nearly rectangular CV shapes for conventional EDLC. The specific capacitance of the active material can be calculated from the charge transferred through the forward and backward scans, which is equal to the area under the curves. The specific capacitance can then be calculated using the following equation^[29]:

$$C = \frac{1}{2 \times v \times \Delta m \times \Delta V} \int I dV \quad (1)$$

where C is the specific capacitance (F g^{-1}), v is the scan rate (V/s), Δm is the mass of the active material (g), ΔV is the potential window (V) and IdV represents the area under CV curve (W). In Fig. 3(c), it can be clearly observed that Co-B@GO exhibits a high specific capacitance of 382 F g^{-1} at 5 mV s^{-1} in 6 mol/L KOH solution, which is higher than those of bare amorphous Co-B alloy (308 F g^{-1}) and GO powder ($\sim 200 \text{ F g}^{-1}$) reported previously^[30, 31]. However, the trends of rate capability curve of the two samples (Co-B@GO and Co-B) are almost the same except that the capacitance of Co-B@GO is higher, which is caused by two main reasons: on the one hand, the layered GO nanosheets in Co-B@GO nanocomposite can provide efficient charge transport channels and enhance the electrolytic accessibility, which improved electrical conductivity. On the other hand, the GO served as a carrier decreases the aggregation and improved utilization efficiency of Co-B nanoparticles^[32, 33], leading to the composite electrode with increasing specific capacitance. It is the synergistic effect of the GO and Co-B nanoparticles, which enhances the electrical performance of nanocomposite.

The high rate discharge capability is crucial for an electrode material to be used efficiently for pseudocapacitor applications. Galvanostatic charge-discharge between -0.2 and 0.5 V was employed to estimate the electrochemical performance of samples as electrode materials for pseudocapacitor. In Fig. 4(a, b), the non-linear charge-discharge curves confirm the pseudocapacitive nature of the Co-B and Co-B@GO electrodes, which results from the electrochemical adsorption/desorption or redox reaction at the electrode-electrolyte interface. The specific capacitance can be calculated from the galvanostatic charge-discharge curve according to Eq. (2)^[34]:

$$C = \frac{I \times \Delta t}{\Delta V \times \Delta m} \quad (2)$$

where C is the specific capacitance (F g^{-1}), I is the constant current (A), Δt is the discharge time (s), ΔV is the potential window (V) and Δm is mass of the electroactive material (g). In Fig. 4(a), the specific capacitance values are 460 F g^{-1} for the Co-B@GO nanocomposites, 300 F g^{-1} for the Co-B nanoparticles. The obtained result is similar to the CV measurement, which also shows the enhanced electrochemical properties after loading the Co-B nanoparticles on the GO. The remarkable enhancement in specific capacitance of the Co-B@GO nanocomposites can mainly be attributed to the high dispersibility of Co-B nanoparticles and high conductivity of GO. As seen in Fig. 4(b), the Co-B@GO nanocomposite has been investigated for galvanostatic charge-discharge study at current

rates of 1, 2, 5 and 10 A g⁻¹ within the potential range of -0.2 to 0.45 V. From Fig. 4(b), we can find that a specific capacitance of 460 F g⁻¹ can be delivered at a current density of 1 A g⁻¹. However, the specific capacitance of Co-B@GO begins to drop as the current increases. This confirms that at the low currents, the ions have enough time to diffuse into the electrode material, while at high currents the ions can only partially penetrate into the electrode material due to the steric limitations^[35]. It is worthwhile mentioning that the composites can still deliver a capacitance of 310 F g⁻¹ even at a very high current density of 10 A g⁻¹, indicating that the composite electrode had a good rate capability.

EIS analysis has been recognized as one of the principal methods to examine the fundamental behavior of electrode materials for pseudocapacitors. Nyquist plots in Fig. 5(a) are composed of an arc in the high frequency region and a nearly straight line in the low frequency region, where Z' and $-Z''$ are real and imaginary parts of the impedance, respectively. As shown in Fig. 5(a) inset, the EIS data can be fitted by an equivalent circuit. In Fig. 5(b), the high frequency intercept with the X axis represents the combined resistance (R_s) or better known as equivalent series resistance (ESR) including the ionic resistance of the electrolyte, the intrinsic resistance of active material, and the contact resistance at the interface of the active material/electrolyte interface. The ESR of Co-B@GO nanocomposite electrode is 0.8359 Ω , which is lower than that of Co-B electrode (2.201 Ω). This indicates that the Co-B@GO nanocomposite electrode has higher electrical conductivity than the bare Co-B nanoparticles. The semicircle represents the charge transfer resistance (R_{ct}) at the electrode/electrolyte interface. The R_{ct} of Co-B and Co-B@GO electrodes are about 0.01733 and 0.01817 Ω , respectively, which are similar to both electrodes. The straight lines in the low frequency region reflect the ion diffusion/transport from the electrolyte to electrode. The phase angles for impedance plots of both electrodes were observed to be higher than 45° in the low frequencies clearly. These findings suggest that both electrodes are not controlled by a diffusion process. The cycling performance, another important index for electrode materials in pseudocapacitors, is shown from the specific capacitance as a function of cycle number based on CV measurement at a scan rate of 10 mV s⁻¹. As shown in Fig. 5(c), the specific capacitance gradually increases in the first 50 cycles, and thereafter, the capacitance begins to decrease. However, after 600 cycles, the specific capacitance becomes stable. After 1000 cycles, the capacitance is still approximately 84% of the first cycle, much higher than that of Co-B/MCNA (60% retained)^[22], which suggests that the GO carrier greatly improves cycling stability compared to MCNA.

4. Conclusion

In conclusion, a nanocomposite of Co–B@GO was prepared by chemical reduction. The GO endows the nanocomposite with high-rate transport of both electrolyte ions and electrons throughout the electrode and high electrochemical utilization of Co–B nanoparticles, resulting in the excellent electrochemical performance. The resulting Co–B@GO nanocomposite achieved a high specific capacitance of 460 F g^{-1} at a current rate of 1 A g^{-1} , which is about 1.5-fold higher than that of the bare amorphous Co–B alloy. Remarkably, up to 67.4% of the capacitance is retained when the charge–discharge rate is increased 10 times from 1 to 10 A g^{-1} , indicating the excellent high-rate properties of the as-synthesized Co–B@GO nanocomposite. The retention of the specific capacitance, based on this initial value after a 1000-cycle test, was 84%, indicating high stability of the electrode for long cycle life. Thus, we believe that it is a promising way to anchor amorphous alloys onto conductive graphene sheets for producing high-performance and high stability electrode materials for energy storage device applications.

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Figure captions

Fig. 1. XRD patterns of CO-B@GO and GO.

Fig. 2. SEM micrograph of GO (a), TEM micrographs of Co-B (b) and CO-B@GO (c, d). The insets in Fig. 2(c, d) shows the SAED patterns of GO and the Co-B@GO nanocomposite, respectively.

Fig. 3. (a) Cyclic voltammetry curves of Co-B and Co-B@GO at a scanning rate of 5 mV s^{-1} . (b) Cyclic voltammetry curves of Co-B@GO electrode at different scan rates. (c) The specific capacitance of Co-B and Co-B@GO at different scan rates.

Fig. 4. (a) The charge-discharge curves of Co-B and Co-B@GO at a current density of 1 A g^{-1} ; (b) the charge-discharge curves of Co-B@GO at different current densities; (c) the specific capacitance of Co-B@GO at various discharge current densities.

Fig. 5. (a) Impedance spectra of Co-B and Co-B@GO (inset is the equivalent circuit), (b) the magnification of the Nyquist curves, (c) cycling performance of Co-B@GO at a scan rate of 10 mV^{-1} . Z' is real impedance and Z'' is imaginary impedance.

Figure List

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5