Construction of NiCo$_2$O$_4$@NiFe LDHs core/shell nanowires array on carbon cloth for flexible, high-performance pseudocapacitor electrodes

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NiFe layered double hydroxides (LDHs), a class of ionic layered compounds with high electrochemical activity, possess great potential in the field of pseudocapacitive energy storage. However, the practical applications of NiFe LDHs in supercapacitors are largely hindered by their inherently poor cycling life and low electrical conductivity. In this work, NiFe LDHs electrodeposited on mesoporous and conductive NiCo$_2$O$_4$ nanowire arrays that are grown on flexible carbon cloth, are investigated as active materials for pseudocapacitive application. This rational core/shell electrode design promises high specific surface area, shorten electron and ion transport distance for the enhancement of electrochemical kinetics. As such, a high specific capacitance (1.9 F cm$^{-2}$ at 1 A cm$^{-2}$, and 1160 F g$^{-1}$ at 1 A g$^{-1}$), excellent rate capability (83% retention as the current density was increased from 1 A cm$^{-2}$ to 20 A cm$^{-2}$), decent cycling stability (79% retention after 1000 charging/discharging cycles), and good flexibility have been obtained, implying their potential application as energy storage devices for flexible electronics.

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1. Introduction

The search for renewable and clean energy supply system has been a pressing challenge facing humanity in recent decades due to the finite availability and adverse environmental impact of traditional energy technologies [1,2]. Supercapacitors, known for their rapid charge-discharge rates, high power densities, and long cycle lives [3], play a key role throughout the renewable energy supply chain, as they are critical to energy conversion, storage, and delivery. In general, supercapacitors can be divided into: (i) electrochemical double layer capacitors (EDLC), which utilizes carbon-based materials (e.g., activated carbon, carbon nanotubes, and graphene) and relies on the accumulation of charges at the electrode-electrolyte interface [4,5], and (ii) pseudocapacitors, which derives their capacitances from fast and reversible Faradic reactions from redox-active materials [6,7]. Among the two types of supercapacitors, pseudocapacitors can offer much higher specific capacitance than EDLC owing to their effective redox-reaction mechanism for energy storage [8], with transition metal oxides, hydroxides, and conductive polymers as typical electrode materials. However, the practical applications of these pseudocapacitive materials are hindered by their poor cycling life and low electrical conductivity [9,10], which largely restricts the achievement of good stability and rate capability.

NiFe LDHs, widely applied in the field of electrocatalysis [11–13], chemical sensing [14,15], and photolytic degradation [16], are a class of ionic lamellar compounds with high electrochemical activity. Their uniquely abundant redox active sites (Ni$^{2+}$, Fe$^{3+}$) and brucite-like layered nanostructures, confer significant enhancement in their pseudocapacitance, even though relevant reports are very limited. Li et al. prepared three dimensional (3D) atomically thin NiFe LDHs microspheres on carbon collector as supercapacitor electrodes by a solvothermal route followed by slurry-drying method, and obtained a capacitance of 1061 F g$^{-1}$ at 1 A g$^{-1}$ [17].
Lu et al. synthesized NiFe LDHs nanosheet directly on nickel foam through a hydrothermal method, which gives a specific capacitance of 2708 F g⁻¹ at 5 A g⁻¹ [18]. Li et al. hydrothermally synthesized flower-like NiFe LDHs on nickel foam, which are next coated with MnO₂ nanosheet. The composite electrode showed a high capacitance at 4.27 F cm⁻² at 5 mA cm⁻² [19]. Although these work demonstrated the usefulness of NiFe LDHs as active charge storage material, suboptimal and overly dense stacking of NiFe LDHs restricted the accessible surface area available for Faradaic charge transfers. The electrically insulating property of stacked NiFe LDHs further limited electron transfer, and which decreased the rate capability and specific capacitance. Furthermore, from a structural standpoint, the use of rigid metal current collector may not be suitable for applications where flexibility is important.

To solve the issues of “low conductivity” and “dead surface”, and thus enable full utilization of active materials, so as to boost the specific capacitance and rate capability, a viable strategy is to engineer pseudocapacitive materials onto a three-dimensional (3D) conductive nanoscale scaffold. Doing so provides increased active surface area, shortened diffusion pathway of electrons and ions, and reliable electrical connections [20,21]. To this end, nanostructured NiCo₂O₄, a spinel binary metal oxide with high Faradaic activity and outstanding electronic conductivity [22], can be an excellent scaffold onto which NiFe LDHs can be readily deposited.

Here, we developed an advanced electrode architecture consisting of NiCo₂O₄ core/NiFe LDHs shell (NiCo₂O₄@NiFe LDHs) nanowire arrays (NWAs) on carbon cloth (CC). The “core” NiCo₂O₄ NWAs are grown via a simple hydrothermal method combined with a post-annealing treatment. Next and importantly, the NiFe LDHs shell is electrodeposited on the NiCo₂O₄ core. A relatively quick NiFe LDHs shell electrodeposition process, as compared to the traditional chemical synthesis methods, provides a more facile and significantly less time-consuming synthesis route. This, to the best of authors’ knowledge, is the first time electrodeposited NiFe LDHs are being applied in pseudocapacitive application. The concept and process is illustrated in Scheme 1. With this rational design, flexible electrode structure, high specific surface area, shortened electron and ion transport pathways can be obtained to produce flexible, high-performance pseudocapacitive electrodes.

2. Experimental section

2.1. Synthesis of NiCo₂O₄ NWAs on CC

CC (W0S1009, CeTech) was cleaned with DI water and ethanol for 15 min, respectively, and dried in the oven. In a typical process, 2 mmol Co(NO₃)₂·6H₂O, 1 mmol Ni(NO₃)₂·6H₂O, and 5 mmol CO(NH₂)₂ were dissolved into 35 mL DI water to form a transparent pink solution. The solution was transferred into a Teflon-lined stainless steel autoclaves before a piece of cleaned CC (3 cm × 2 cm) is immersed in it. Next, the Teflon-lined stainless steel autoclave was maintained at 120 °C for 12 h to facilitate hydrothermal growth. After the process ended, the CC sample was rinsed several times with DI water and ethanol under ultrasonication. Finally, the sample was calcined at 300 °C for 2 h and the well-defined NiCo₂O₄ NWAs was formed on the CC. On average, an areal density of ca. 1.5 mg cm⁻² NiCo₂O₄ nanowires was achieved.

2.2. Electrodeposition of NiFe LDHs

First, 10 mmol Fe(NO₃)₃·9H₂O and 40 mmol Ni(NO₃)₂·6H₂O were added into 50 mL DI water to from a transparent green electrolyte solution. Second, the NiFe LDHs were electrochemically reduced and deposited onto the surface of NiCo₂O₄ nanowires at a constant potential of −1.2 V (vs. reference electrode) for 90 s using a three-electrode system. Finally, the sample (known as NiCo₂O₄@NiFe LDHs NWAs) was washed with DI water and dried. On average, the areal density of all active materials, including NiCo₂O₄ NWAs and NiFe LDHs, is 1.7 mg cm⁻². Separately, NiFe LDHs were electrodeposited onto a pristine CC (known as CC@NiFe LDHs) following the same process as a reference specimen, the electrodeposited NiFe LDHs loading is ca. 0.1 mg cm⁻².

2.3. Materials characterization

The morphology and structure of as-prepared samples were observed with a field-emission scanning electron microscope (FESEM; Gemini SEM 500, Zeiss, 15 kV). Elemental characterization was conducted with an energy dispersive X-ray spectrometer (EDS) through areal elemental mapping and analysis. The mesoporous structure, high-resolution transmission electron micrographs (HRTEM), and selected area electron diffraction (SAED) were recorded with a transmission electron microscope (TEM; JEM-F200, JEOL). The crystal structure was characterized by X-ray diffraction (XRD; D8 Advanced, Bruker) using Cu Kα radiation over a 2θ range of 10°–70°. Raman spectroscopy measurements were performed on a micro–Raman spectroscopy system (HR8000, Horiba Scientific) with a 514.5 nm laser excitation. X-ray photoelectron spectrums (XPS) were obtained by a photoelectron spectrometer (ESCALAB...
2.4. Electrochemical measurements

Electrochemical characterization of the as-synthesized samples were conducted using a Gamry Reference 600 + electrochemical workstation in an aqueous KOH electrolyte (2.0 M) with a three-electrode cell. The as-prepared samples were used as working electrode without any further treatment. Pt foil and Ag/AgCl electrode serve as the counter electrode and the reference electrode, respectively. Cyclic voltammetry (CV) tests were obtained in the potential range between 0 V and 0.45 V by varying the scan rates from 2 to 20 mV s$^{-1}$. Galvanostatic charge-discharge (GCD) experiments were performed with different current densities ranged from 1 mA cm$^{-2}$ to 20 mA cm$^{-2}$ (and 1 A g$^{-1}$ to 20 A g$^{-1}$) within a 0–0.4 V potential window. Electrochemical impedance spectroscopy (EIS) were carried out with the frequency range from 10$^5$ Hz to 10$^{-2}$ Hz using a perturbation amplitude of 5 mV at 0.2 V versus reference electrode. Cycling performance was tested for 1000 cycles at a current density of 15 A g$^{-1}$.

3. Results and discussion

3.1. Morphology and structure

Large-area, uniform, and dense NiCo$_2$O$_4$ NWAs grew radially outward on individual carbon fiber in the CC following the hydrothermal synthesis (Fig. 1a and Fig. S1a). Each single nanowire has a diameter of ca. 50 nm and length of up to several micrometers (Fig. 1b and Fig. S1b). The macroscopic architecture of the 3D oriented carbon fibers and the large open spaces between adjacent nanowires can promote better penetration of the electrolyte. After electrodeposition, the NiCo$_2$O$_4$ nanowires were decorated uniformly with NiFe LDHs (Fig. 1c). The NiFe LDHs-coated NiCo$_2$O$_4$ NWs samples can be clearly distinguished from the bare NiCo$_2$O$_4$ NWs, as evident from the increased diameters (ca. 60–80 nm) and significantly higher surface roughnesses of the NiCo$_2$O$_4$@NiFe LDHs NWs (Fig. 1d). Despite observable agglomeration of NiCo$_2$O$_4$@NiFe LDHs NWs, it is worth noting that CC samples coated with NiCo$_2$O$_4$@NiFe LDHs NWAs still exhibit much higher specific area compared to CC coated with NiFe LDHs alone (Fig. S2). The EDS elemental mapping, as shown in Fig. 1e, clearly demonstrates the homogeneous distribution of O, Co, Ni, Fe elements over the electrode sample.

The morphologies and structures of the samples were further examined using TEM. The NiCo$_2$O$_4$ nanowire is a mesoporous nanostructure, as shown in Fig. 2a, composed of 10–20 nm nanocrystallites with pores of ca. 2–8 nm in diameters. This mesoporous property of NiCo$_2$O$_4$ nanowires can facilitate fast charge transport and ion diffusion [23]. The lattice spacing of ca. 0.47 nm observed in Fig. 2b is in good agreement with previous report for NiCo$_2$O$_4$ [24]. Fig. 2c shows a TEM image of one individual NiCo$_2$O$_4$@NiFe LDHs core/shell nanowire, in which the “core” NiCo$_2$O$_4$ nanowire and the “shell” NiFe LDHs (red arrow) can be clearly distinguished, demonstrating the successful deposition of NiFe LDHs onto the surface of NiCo$_2$O$_4$ nanowires. The hierarchical nanostructure of NiCo$_2$O$_4$@NiFe LDHs core/shell nanowire was further confirmed by HRTEM as shown in Fig. 2d. It can be clearly seen that the NiFe LDHs

Fig. 1. SEM images of (a, b) NiCo$_2$O$_4$ NWAs on one single carbon fiber, (c, d) NiCo$_2$O$_4$@NiFe LDHs NWAs on one single carbon fiber. (e) EDS elemental mapping of O, Co, Ni, Fe elements throughout one single NiCo$_2$O$_4$@NiFe LDHs NWA.
layer are composed of crystalline (marked with red arrows) and amorphous structure, proving the semicrystalline structure of electrodeposited NiFe LDHs. For the lattice part, the fringe spacing of ca. 0.26 nm agrees well with the spacing lattice plane (012) of NiFe LDHs [25], further demonstrating the successful deposition of NiFe LDHs on NiCo2O4 nanowires.

3.2. Materials characterization

The EDS spectrum of NiCo2O4@NiFe LDHs core/shell NWAs on CC, as illustrated in Fig. 3a, provides the clear evidence of the presence of O, Co, Ni, and Fe elements. The Ni: Co ratio of the as-obtained samples is ca. 5.55:1 (Table S1), which is higher than that of the electrodeposition electrolyte precursor (4:1). This should be attributed to the Ni element contained from NiCo2O4 nanowires scaffold.

The XRD patterns were detected and shown in Fig. 2b. In the XRD pattern of CC@NiFe LDHs sample (black line), three small peaks at 2θ = 11.7°, 33.6°, 60° are observed, and these peaks correspond to (003), (012), and (110) crystallographic planes of α-Ni(OH)2, respectively [25]. The amplitude of these peaks are relatively weak probably due to the crystallinity of the electrodeposited NiFe LDHs is limited. The XRD pattern of NiCo2O4 nanowires (blue line) reveals a cubic crystal structure. The mean crystallite size of NiCo2O4 is calculated as ca. 12 nm using the Scherrer equation, implying the presence of quasi-continuous nano-subunits within NiCo2O4 nanowires, already corroborated by TEM observations. Upon electrodeposition of NiFe LDHs onto the surface of NiCo2O4 NWAs, all the peaks of NiFe LDHs and NiCo2O4 nanowires show up synchronously (red line), confirming the successful synthesis of NiCo2O4@NiFe LDHs composites.

Furthermore, Raman spectroscopy was utilized to characterize the phase composition of the as-prepared samples. As seen from Fig. 2c, the characteristic peaks of CC@NiFe LDHs are near 530 cm⁻¹ and 700 cm⁻¹, which are consistent with previous report [26]. The peaks at 186, 456, 504, and 648 cm⁻¹ corresponded to the F₂g, E₂g, F₂g, A₁g modes of the NiCo2O4 nanowires, respectively [20]. Based on the Raman spectrum of the NiCo2O4@NiFe LDHs core/shell NWAs, the observed bands shift and intensity enhancement indicate strong coupling interaction between NiCo2O4 nanowires and NiFe LDHs.

The more detailed elemental composition and chemical state were further characterized by XPS, as illustrated in Fig. 3d–f. In the full scan from 0 to 1000 eV, the existence of O, Co, Ni, and Fe elements is observed in Fig. 3d. The high resolution of Ni 2p spectrum (Fig. 3e) shows two peaks at 855.2 and 873 eV, which can be identified as Ni 2p₃/₂ and Ni 2p₁/₂, respectively, along with two satellite peaks (denoted as “Sat”), implying the valence of Ni is 2⁺ [27]. In the case of Fe 2p, a pair of peaks at 711.3 and 724.3 eV corresponding to Fe 2p₃/₂ and Fe 2p₁/₂ can be seen from Fig. 3f, indicating the Fe³⁺ oxidation state in the NiFe LDHs [28]. The results are in good agreement with characterization results from previous work [25], where electrodeposited NiFe LDHs were used as catalysts for water splitting. The above XPS analyses reveal the electrodes are composed of enriched highly redox-active Ni²⁺ and Fe³⁺ ions, which are of great importance for effective pseudocapacitive reactions.

3.3. Electrochemical performance

Fig. 4a illustrates the CV curves of CC@NiFe LDHs, NiCo2O4 NWAs, and NiCo2O4@NiFe LDHs core/shell NWAs at the scan rate of 10 mV s⁻¹. It can be clearly seen that the NiCo2O4@NiFe LDHs core/shell NWAs electrodes exhibit a substantially larger area as compared to that of the CC@NiFe LDHs and NiCo2O4 NWAs samples. This highlights the superior pseudocapacitive performance of the
composite electrode. GCD measurements were performed on these three electrodes at a current density of 1 mA cm\(^{-2}\). As illustrated in Fig. 4b, the discharge time of NiCo\(_2\)O\(_4@\)NiFe LDHs core/shell NWAs is ca. 760 s, which is 6.5 times and 5.2 times higher than that of CC@NiFe LDHs (ca. 116 s) and NiCo\(_2\)O\(_4\) NWAs (ca. 145 s), respectively, showing significant enhancement of the specific capacitance of the advanced NiCo\(_2\)O\(_4@\)NiFe LDHs core/shell NWAs electrodes. Fig. 4c shows the typical CV curves of NiCo\(_2\)O\(_4@\)NiFe LDHs core/shell NWAs at different scan rates that ranged from 2 mV s\(^{-1}\) to 20 mV s\(^{-1}\). A pair of well-defined redox peaks can be clearly observed, indicating the presence of pseudocapacitive charge storage on the as-prepared electrodes. Apparently, with the increase of scan rate, the peak value increases, and the cathodic and anodic peaks shift towards lower and higher potential, respectively. All these behaviors demonstrate the rapid electronic and ionic transport rate of this novel electrode.

Fig. 3. (a) EDS results of the NiCo\(_2\)O\(_4@\)NiFe LDHs core/shell NWAs. (b) XRD pattern and (c) Raman spectrum of CC@NiFe LDHs, NiCo\(_2\)O\(_4\) NWAs, and NiCo\(_2\)O\(_4@\)NiFe LDHs core/shell NWAs. (d) XPS spectrum of the NiCo\(_2\)O\(_4@\)NiFe LDHs core/shell NWAs, and the high resolution spectra of (e) Ni 2p, (f) Fe 2p.

Fig. 4. (a) CV and (b) GCD curves of CC@NiFe LDHs, NiCo\(_2\)O\(_4\) NWAs, and NiCo\(_2\)O\(_4@\)NiFe LDHs core/shell NWAs at constant scan rate (10 mV s\(^{-1}\)) and constant current density (1 mA/cm\(^2\)), respectively. (c) CV and (d) GCD of NiCo\(_2\)O\(_4@\)NiFe LDHs core/shell NWAs under different scan rates and current densities, respectively. (e) rate capability, area-normalized specific capacitance as a function of current density. (f) cycling stability tested at current density of 15 A g\(^{-1}\).
To evaluate the rate capability, the GCD curves of NiCo2O4@NiFe LDHs core/shell NWAs at different current density from 1 mA cm\(^{-2}\) to 20 mA cm\(^{-2}\) in the potential range of 0–0.4 V were measured as illustrated in Fig. 4d, and the area-normalized specific capacitance were calculated and recorded in Fig. 4e (red line with star). NiCo2O4@NiFe LDHs core/shell NWAs exhibit excellent areal capacitance of 198, 189, 184, 178, 172, and 163 F cm\(^{-2}\) when subjected to current densities of 1, 2, 4, 8, 12, and 20 A cm\(^{-2}\), respectively. For NiCo2O4@NiFe LDHs core/shell NWAs, a remarkable 83% of areal capacitance was retained when the current density increased from 1 A cm\(^{-2}\) to 20 A cm\(^{-2}\). The GCD of CC@NiFe LDHs and NiCo2O4 NWAs were also measured (Fig. S3). Compared with NiCo2O4@NiFe LDHs core/shell NWAs, only 17.4% (black line with circle in Fig. 4e) and 76% (blue line with square in Fig. 4e) are retained for CC@NiFe LDHs and NiCo2O4 NWAs, respectively. Gravimetric capacitances were measured and recorded in Fig. S4. NiCo2O4@NiFe LDHs core/shell NWAs shows a gravimetric capacitance of 1160 F g\(^{-1}\) at 1 A g\(^{-1}\) and 850 F g\(^{-1}\) at 20 A g\(^{-1}\), demonstrating a gravimetric capacitance retention of 73%. In comparison, only 50.3% and 65% of gravimetric rate capability of the NiCo2O4@NiFe LDHs core/shell NWAs electrodes presented in this work.

The cycling performances of the as-prepared samples were evaluated by the repeated charging/discharging at a constant current density of 15 A g\(^{-1}\) in the potential range of 0–0.4 V for 1000 cycles, as illustrated in Fig. 4f. The NiCo2O4 NWAs shows the highest cycling stability with a high gravimetric capacitance retention of ca. 91%. The CC@NiFe LDHs exhibits the worst cycle life, only ca. 58% capacitance is retained due to the serious agglomeration and abscission of the NiFe LDHs on the CC substrate (Fig. S5). The NiCo2O4@NiFe LDHs core/shell NWAs displayed a decent capacitance retention of ca. 79% after 1000 cycles. SEM observations were performed to investigate the causes of the performance loss during cycling. As illustrated in Fig. S6, after 1000 charging/discharging test, a substantial portion of vertically grown nanowires had either agglomerated or collapsed, lowering the number of NiFe LDHs that can effectively participate in the Faradaic reactions. It is worth mentioning that no visible abscission of NiFe LDHs from the surface of NiCo2O4 NWAs was observed, pointing to the robust adhesion of NiFe LDHs on NiCo2O4 NWAs compared to that of NiFe LDHs directly grown on CC substrate.

The above electrochemical measurements and analyses indicate that, compared with CC@NiFe LDHs and NiCo2O4 NWAs, the electrodes made up of NiCo2O4@NiFe LDHs core/shell NWAs on CC substrate deliver the highest areal and gravimetric capacitances, excellent rate capability, and decent cycling stability. These high performance features can be ascribed to the advanced electrode architecture design in the following ways: (i) the three-dimensional NiCo2O4@NiFe LDHs core/shell NWAs structure with a much higher specific surface area, combined with the large open spaces between adjacent nanowires, are of great advantage for efficient electrolyte penetration, enabling high utilization of NiFe LDHs. Moreover, at some degree, the vertically grown NWAs architecture prevents the agglomeration and abscission of NiFe LDHs. (ii) excellent conductivity and mesoporous properties of NiCo2O4 NWAs skeleton promote fast charge transfer and ion diffusion, dramatically decreasing the interfacial charge-transfer resistance (EIS analysis, see Fig. S7 and discussions), and thus enhancing the electrochemical kinetics.

To investigate the mechanical flexibility of the as-prepared electrodes, a sample was bent or twisted into an arbitrary shape by a pair of tweezers (Fig. S8a), and it can spring back to the original shape after the force was removed without any visible permanent deformation or damage. As illustrated in Fig. 5a, the CV curves show negligible changes when the electrodes was bent at the angles of 30°, 90°, 180° or twisted into a S-shaped structure. In addition, a home-made linear actuator (inset of Fig. 5b) was utilized to induce repeat mechanical bending to the sample. As seen from Fig. 5b, the capacitance retention was measured to be about 99%, 97%, and 94% (GCD measurements, see Fig. S8b) after the sample was repeatedly bent for 300, 500, and 1000 times at 30°, respectively. All these tests highlight the excellent flexible performance and durability of our electrodes.

4. Conclusion

In summary, NiCo2O4@NiFe LDHs core/shell NWAs directly grown on CC were developed as electrode for flexible, high performance pseudocapacitors. The advanced electrode architecture simultaneously demonstrated the advantageous combination of flexible electrode structure, efficient mass transport of electrolyte, high electron transfer rate, fast ion diffusion rate, as well as low interfacial electron-transfer resistance. As such, the NiCo2O4@NiFe LDHs core/shell NWAs exhibited excellent pseudocapacitive behaviors, including a high specific capacitance, a good rate capability, and a decent cycling stability, implying their potential application as energy storage devices for flexible electronics.
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Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.jallcom.2018.07.188.

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