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Electrochemically Synthesized Nanoflowers to Nanosphere-Like NiCuSe₂ Thin Films for Efficient Supercapacitor Application

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Received: 16 November 2020; Accepted: 17 December 2020; Published: 21 December 2020



Abstract: Developing efficient electrochemically active nanostructures from Earth-abundant elements has gained significant interest in recent years. Among different transition metals, nickel and copper are abundant electrocatalysts for energy-storage applications. Nickel–copper selenide (NiCuSe₂) nanostructures were prepared on a stainless-steel mesh with a cost-effective, simple, and versatile electrodeposition method for supercapacitor applications. The change effect in the bath concentration of nickel and copper altered the structural and electrochemical properties of NiCuSe₂ electrode. X-ray diffraction (XRD) patterns confirmed the pure phase of ternary NiCuSe₂ thin films with a cubic crystal structure. The surface morphology of NiCuSe₂ was tuned by nickel and copper from spherical porous nanoflowers, nanoplates, nanocubes, and nanosphere-like nanostructures deposited on the stainless-steel mesh. The electrochemical performance of the electrodeposited NiCuSe₂ was investigated in alkaline 1 M KOH electrolyte. The synergetic effect of bimetallic nickel and copper with the selenide electrode showed superior specific capacity of about 42.46 mAh g⁻¹ at 10 mV s⁻¹ along with reasonable cycling stability.

Keywords: nickel–copper selenide; electrodeposition; nanostructures; supercapacitor; stainless-steel mesh; nanoflakes

1. Introduction

The rapid depletion of fossil fuels and rising pollution have led to high demand for alternative energy-harvesting and -storage systems [1]. Supercapacitors are an advanced type of electrochemical energy-storage technology [2,3], which is used in various electronic devices, electric vehicles, solar cells, and wearable devices. Supercapacitors have advantages such as high power density, high energy density, long life cycle, and low cost of manufacturing and maintenance [4,5]. Recently, porous carbons, conducting polymers, and several transition-metal compounds composed of oxides, nitrides, sulfides, and selenides have been utilized as electrodes for electrochemical-energy storage applications [6–9]. The poor electrical conductivity of transition metal oxides/hydroxides and the lower stability of sulfides limit their electrochemical performance [10]. Among these materials, transition-metal selenides are promising electrode materials for supercapacitors, oxygen evolution, and other applications because of their good electrical conductivity, higher values of theoretical specific capacity, and structural and optoelectronic properties [11–13].



Metal selenides, made up of Cu, Bi, Sn, etc., have low band-gap energy and are applied in photoelectrochemical cells for solar-energy harvesting systems and electrocatalytic applications [13,14]. Similarly, binary metal selenides were synthesized and are devoted to improving electrocatalytic activity. Recently, bimetallic Cu–Co selenide nanowires showed improvement in the electrochemical properties of supercapacitors [15]. Tang et al. fabricated NiSe nanowires on nickel foam via a two-step hydrothermal method, which exhibited capacitance of 1790 F g⁻¹ at 5 A g⁻¹ [16]. Zhang et al. reported stable SnSe nanosheets having energy-storage capacity (228 F g⁻¹ at 0.5 A g⁻¹) using a high-temperature refluxing method [17]. Furthermore, Chen et al. fabricated a Ni–Co selenide supercapacitor by a hydrothermal method, exhibiting a specific capacity of 535 C g⁻¹ at 1 A g⁻¹ [18]. These methods involve complex processes and are not binder-free electrode-preparation approaches. Binder-containing electrodes also have poor electrical conductivity and cause an increased dead surface during electrochemical studies [10].

Transition-metal selenides are produced by various methods, including chemical bath deposition [19], evaporation, and electrodeposition [20]. Among these methods, electrodeposition has advantages such as low cost, ease to control nanostructure growth, simple, and ecofriendly. Appropriate metals for bimetallic selenides are nickel $(1.43 \times 107 \text{ S m}^{-1})$ and copper $(5.96 \times 107 \text{ S m}^{-1})$ because of their high electrical conductivity compared to that of other metals, which might assist to preserve a higher electrical conductivity for selenium $(1 \times 10^{-3} \text{ S m}^{-1})$.

In this study, we report an electrodeposition method for the preparation of nickel–copper selenide nanostructures. Some reports were on the preparation of ternary NiCuSe₂ via other chemical methods. However, less attention has been paid towards electrodeposition and supercapacitor applications. We developed various NiCuSe₂ coated nanostructures on stainless-steel mesh by a potentiostatic mode of the electrodeposition method. The effect of nickel and copper bath concentration with a constant concentration of selenide on the structural and electrochemical properties was studied. The prepared electrodes were used for the measurements of supercapacitive properties in the 1 M KOH alkaline electrolyte.

2. Experiment Details

2.1. Chemicals and Materials

Nickel sulfate hexahydrate (NiSO₄· $6H_2O$), copper sulfate anhydrous (CuSO₄), and selenium dioxide (SeO₂) were procured from Sigma-Aldrich, Seoul, Korea. Potassium hydroxide (KOH) was purchased from Daejung, Gyeonggi-do, Korea. All chemicals were of analytical grade and used as received.

2.2. Synthesis of NiCuSe₂ Thin Films

NiCuSe₂ nanostructures were deposited by an electrodeposition method on the stainless-steel mesh. The electrochemical bath contained an aqueous solution of nickel sulfate hexahydrate, copper sulfate, and selenium dioxide in a separate beaker. Deposition was carried out on a cleaned stainless-steel mesh at room temperature with a potentiostatic mode at -0.75 V for 120 s constant for all samples. The molarity effect of the nickel and copper precursor on the electrochemical properties was investigated. The molarity of nickel and copper was varied, while selenium molarity was kept constant throughout the experiment. The four samples were obtained with nickel concentrations of 0.0025, 0.0050, 0.0075, and 0.01 M, and copper concentrations of 0.011, 0.008, 0.004, and 0.015 M, and denoted as S-1, S-2, S-3, and S-4, respectively.

2.3. Characterizations

Field-emission scanning electron micrographs (FE-SEM) were recorded on an JEOL microscope (JEOL JSM-7100, Tokyo, Japan). Powder X-ray diffractograms (XRD) were recorded with a D8 advanced diffractometer system (Bruker, Billerica, MA, USA) equipped with a Cu–K $_{\alpha}$ radiation source (40 kV,

40 mA). X-ray photoelectron spectra (XPS) were collected with an ESCALABMK II X-ray photoelectron spectrometer (Thermo Fisher Scientific, Waltham, MA, USA) equipped with an Mg–K_{α} X-ray source. Electrochemical analysis was performed using a VersaSTAT 3 Potentiostat Galvanostat workstation (Princeton Applied Research, Princeton, NJ, USA).

2.4. Electrochemical Measurements

Cyclic-voltammetry (CV) and galvanostatic-charge/discharge (GCD) measurements were conducted on a VersaSTAT 3 electrochemical workstation using a typical three-electrode system at room temperature. Stainless-steel mesh was used as working electrode, while a Pt wire and a saturated Ag/AgCl electrode served as the counter and reference electrode, respectively. Electrochemical characteristics were studied in aqueous 1 M KOH solution, and the geometric area of the working electrode was one unit cm² for all electrodes.

3. Results and Discussion

3.1. NiCuSe₂ Characterization

Figure 1 presents the XRD patterns of all NiCuSe₂ samples prepared by the potentiostatic mode of the electrodeposition method. A strong diffraction peak of the stainless-steel mesh was observed at 43.54° for all NiCuSe₂ samples. Diffraction peaks of NiCuSe₂ at 36.42° and 50.66 were consistent with the (211), (311), and (332) planes of cubic NiCuSe₂ (JCPDS 00-018-0888). The diffraction peaks of Cu at 50.66° and 74.4° overlapped with the peak of Ni, and matched with the (200) and (220) planes of cubic Cu (JCPDS 00-004-0836) and cubic NiCuSe₂ (JCPDS 00-006-0507). The peak at 36.42° was only observed for S-1 due to the formation of defects with the higher concentration of copper [21]. These results confirmed the formation of pure ternary phase of the NiCuSe₂. The intensity of the nickel peak was linearly increased, while the intensity of the steel peak was linearly decreased from S-1 to S-4. The intensity of nickel and copper peaks was altered with the concentration variation experiment without any shift in peak positions. The crystallite sizes of the samples were calculated using the Scherrer equation: 31.47, 32.62, 32.74, and 33.85 nm for samples S-1, S-2, S-3, and S-4, respectively.



Figure 1. XRD patterns of all samples.

FE-SEM images for all samples with different magnifications are shown in Figure 2. NiCuSe₂ nanostructures were clearly uniformly deposited on the stainless-steel mesh. Sample S-1 showed highly porous spongy nanoflake-like nanostructures deposited on the stainless-steel mesh. After changing bath concentration, interconnected nanoflake-like nanostructures grew on the surface of the steel mesh. This type of surface morphology provided more surface area during electrochemical testing. Further, by changing the bath concentration of Ni and Cu, the surface of NiCuSe₂ showed a regular deposition of a nanocubic-like nanostructure developing on the surface of the stainless-steel mesh. However, at a higher bath concentration of Ni and Cu ions, there was a drastic change in surface morphology from the cubic to the nanosphere. Spongy nanoflake, interconnected nanoflakes, nanocubic, and nanosphere-like morphology was observed in all samples, while the size, thickness, and length of the nanostructures was altered with the change in electrolyte bath concentration of Cu and Ni ions for ternary NiCuSe₂ [22–24]. Nanoflake length for sample S-2 was slightly higher than that of the other samples, while nanosphere size for sample S-4 was comparatively higher than that of other samples [25,26].



Figure 2. FE-SEM images of Samples (a-c) S-1, (d-f) S-2, (g-i) S-3, and (j-l) S-4.

XPS analysis was performed to further study the chemical environment of NiCuSe₂. The representative XPS spectrum of sample S-4 is depicted in Figure 3. Figure 3a shows the survey spectrum of S-4, which exhibited peaks of nickel, copper, selenide, carbon, and oxygen without any other impurities. Peaks of C 1s and O 1s were detected for the sample due to exposure to air (Figure 3b,c). The spectrum of Cu 2p confirmed that the two main peaks (952.21 and 932.35 eV) could be assigned for Cu $2p_{1/2}$ and Cu $2p_{3/2}$, respectively (Figure 3d). Similarly, peaks at 855.61 and 873.27 eV appeared due to Ni $2p_{1/2}$ and Ni $2p_{3/2}$, respectively. The two corresponding satellite peaks were observed at binding energies of 879.30 and 860.99 eV (Figure 3e). Selenium (Figure 3d) showed peaks at 53.49 and 54.38 eV due to $3d_{5/2}$ and $3d_{3/2}$, respectively (Figure 3f) [27].



Figure 3. XPS of sample S-4. (a) Survey spectrum. Core levels of (b) C 1s, (c) O 1s, (d) Cu 2p, (e) Ni 2p, and (f) Se 3d.

3.2. Electrochemical Study

The electrochemical measurement of the all samples was performed in 1 M KOH solution against Hg/HgO reference electrode at a potential window of 0–0.5 V. Figure 4a shows the CV, and the inset shows the specific capacity of all four samples in a potential range of 0–0.5 V (vs Hg/HgO) at a constant scan rate of 10 mV s⁻¹. The CV curves of S-1, S-2, and S-3 with difference scan rate are depicted in the Figure S1a-c. A well-defined redox peak was observed for S-4 compared to that of the other samples. The specific capacity of all samples was calculated from this CV profile. The specific capacity (Cs) was estimated from $C_s(mAhg^{-1}) = \int I(V)dv/mv \times 3.6$. The specific capacity values for samples S-1 to S-4 were 4.30, 10.84, 22.37, and 42.46 mAh g^{-1} , respectively. The performance of NiCuSe₂ could be optimized by changing the electrolyte bath concentration of Ni and Cu ions. The optimal concentration of Ni and Cu ions in the electrolyte bath was responsible for the higher capacity values of S-4, as shown in the Figure 4b. Figure 4c shows the specific capacity of the optimized NiCuSe₂ electrode with various scan rates, in the range of 10–100 mV s⁻¹, respectively. The obtained values of specific capacity were 42.46, 37.94, 27.36, 21.01, 17.03, and 13.88 mAh g^{-1} for scan rates 10, 20, 40, 60, 80, and 100 mV s^{-1} , respectively. Owning to the excellent supercapacitor performance and higher area of CV for S-4, the sample was selected as a representative to demonstrate the electrochemical properties. Figure 4d shows the GCD curve of S-4 with different current densities. Specific capacity that was estimated from the GCD curve of S-4 was 17.36 mAh g^{-1} at 0.25 mA cm⁻². GCD curves at different current densities were also mainly symmetrical, revealing the good electrochemical reversibility of S-4, as shown in Figure 4d.



Figure 4. (a) Cyclic-voltammetry (CV) curves, inset shows specific capacity for samples S-1, S-2, S-3, and S-4 at 10 mV s⁻¹. (b) CV of S-4 with respect to different scan rates in the range 10–100 mV s⁻¹. (c) Specific capacity of S-4 with different scan rates. (d) Galvanostatic charge/discharge (GCD) of S-4 with different current densities.

The EIS of all samples is shown in Figure 5. The diffusion resistance of electrolyte ions can be seen in the low-frequency region. The straight line in the low-frequency region shows the low diffusion resistance of ions from the KOH solution to the electrode interface. The cycling stability of S-4 up to 1000 cycles was investigated with GCD at 1 mA cm⁻² (as shown in Figure S1d). Sample S-4 maintained reasonable cycling stability, and was slightly decreased in capacitance after 100 cycles due to the irreversible reaction of the material.



Figure 5. Nyquist plots of samples S-1 to S-4.

4. Conclusions

Variation in the electrolyte bath concentration of nickel and copper improved the electrochemical performance of the nickel–copper selenide electrode. The electrochemical study indicated a superior performance for S-4, with a 42.46 mAh g⁻¹ at 10 mV s⁻¹ in 1 M KOH electrolyte. Reasonable cycling stability due to the stable structure on the stainless-steel mesh substrate was also observed. These results proved that the nickel–copper selenide with an optimal concentration of nickel and copper in the electrolyte bath may be a useful candidate for next-generation hybrid supercapacitors and electrocatalytic applications.

Supplementary Materials: The following are available online at http://www.mdpi.com/2075-4701/10/12/1698/s1, Figure S1: (a–c) CV curves of the S-1, S-2 and S-3 samples at a different scan rate from 10–100 mV s⁻¹ in the potential range 0 to 0.5 V with 1 M KOH electrolytes, respectively, (d) cycling stability with respect to the number of cycles.

Author Contributions: Conceptualization, methodology, writing—review and editing, S.K.S. and H.M.Y.; formal analysis, V.G.P. and H.-H.P.; supervision, D.-Y.K. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Acknowledgments: H.M.Y. and S.K.S. are thankful to the Dongguk University for supporting research in 2018-22. V.G.P. and H.-H.P. would like to thank that this work was supported by the National Research Foundation of Korea (NRF), grant funded by the Korean government (MSIT; No. 2020R1A5A1019131).

Conflicts of Interest: The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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