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Citation for final published version:

Pawar, S.M., Pawar, B.S., Hou, Bo , Ahmed, A.T.A., Chavan, H.S., Jo, Yongcheol, Cho, Sangeun, Kim, Jongmin, Seo, Jiwoo, Cha, SeungNam, Inamdar, A.I., Kim, Hyungsang and Im, Hyunsik 2019. Facile electrodeposition of high-density CuCo₂O₄ nanosheets as a high-performance Li-ion battery anode material. *Journal of Industrial and Engineering Chemistry* 69 , pp. 13-17. 10.1016/j.jiec.2018.09.042

Publishers page: <http://dx.doi.org/10.1016/j.jiec.2018.09.042>

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Facile electrodeposition of high-density CuCo_2O_4 nanosheets as a high-performance Li-ion battery anode material

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Abstract

High-density CuCo_2O_4 nanosheets are grown on a nickel foam (NF) substrate using an electrodeposition method followed by air annealing for use as a lithium-ion (Li-ion) battery anode material. It exhibits a high discharge capacity of 1244 mA/g at 0.1 A/g with a coulombic efficiency of 82 %. The CuCo_2O_4 nanosheet anode shows excellent high-rate performance and stability with 95% capacity retention (1100 mAh/g) at 1 A/g after 200 cycles. The superior electrochemical performance of the CuCo_2O_4 nanosheet electrode is attributed to the binder-free direct contact of the electrode active material to the current collector and unique high-density nanosheet morphology.

Keywords: CuCo_2O_4 nanosheets, electrodeposition, Li-ion battery, cyclic voltammetry, anode material

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5 **Introduction**
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8 The increasing concern over energy crisis and environmental pollution from burning
9 fossil fuels has significantly driven the development of clean and renewable energy sources [1].
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12 Among the various energy storage devices, regardless of lithium (Li) scarcity, rechargeable Li-
13 ion batteries (LIBs) are still considered as the most efficient energy storage device at the present
14 time, due to their high energy density, long-term stability, and environmental friendliness [1-2].
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16 For commercially available LIBs, graphite is commonly used as an anode material due to its low
17 price, high conductivity, and high reversibility [3]. However, it has a low theoretical capacity
18 (372 mAh/g) and serious safety issues have arisen from Li dendrite formation, which limits the
19 large-scale application [4]. Therefore, considerable attention is being paid to explore alternative
20 anode materials with high capacity along with excellent cycling stability and rate capability.
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24 Recently, many ternary transition metal oxides including NiCo_2O_4 , CuCo_2O_4 , and
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26 ZnCo_2O_4 have been extensively studied for use as LIB anode materials due to their low cost,
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28 high specific capacity and good stability. The observed high performance of the materials is
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30 understood in terms of the synergetic effects between the two constituting transition metal
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32 elements during the Li^+ discharge/charge process [2,3]. Among these, more attention has been
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34 paid to spinel copper cobaltite (CuCo_2O_4) because of its abundance and environmental
35
36 friendliness. Various nanostructured CuCo_2O_4 anode materials ranging from nanoparticles,
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38 hollow spheres, nanorods, and nanocubes have been reported [5-12]. Table 1 reports the LIB
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40 performance of various nanostructured CuCo_2O_4 . Two-dimensional (2-D) nanostructures exhibit
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42 a better electrochemical performance than other nanostructures due to short Li^+ diffusion
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44 pathways, large electrochemically active sites and low diffusive resistance at the
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5 electrode/electrolyte interface [13,14]. In order to enhance the anode performance of CuCo_2O_4 , it
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7 is desirable to fabricate nanostructured 2-D CuCo_2O_4 directly deposited on the current collector.
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10 In this work, we report the facile electrodeposition of CuCo_2O_4 nanosheets on a nickel
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12 foam (NF) substrate for use as a binder-free anode for LIB. The CuCo_2O_4 nanosheet anode
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14 exhibits a high specific capacity of ~ 1244 mAh/g at 0.1 A/g along with excellent cycling stability
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16 and high rate capability.
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22 **Experimental**

23 **Fabrication of CuCo_2O_4 nanosheet film**

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25 A copper-cobalt hydroxide ($\text{Cu-Co}(\text{OH})_2$) precursor film was prepared on NF substrate
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27 using an electrodeposition method at room temperature. The precursor solution was prepared
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29 using 2.5 mM copper nitrate ($(\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O})$) and 10 mM cobalt nitrate ($(\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$)
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31 solutions. Analytical reagent grade chemicals were used for the precursor solution preparation
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33 (supplied by Sigma-Aldrich). Prior to the electrodeposition, the NF substrate was cleaned with a
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35 3 M HCl solution using ultra-sonication for 10 min to remove the nickel oxide layer. The NF
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37 substrate was washed with acetone, ethanol and distilled water for 10 min and then it was dried
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39 at 60 °C for 24 h in the air environment. The electrodeposition was carried out using a
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41 conventional three-electrode cell where a Pt wire was used as the counter electrode, while the
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43 saturated calomel electrode (SCE) and an NF substrate with an area of 2×4 cm² served as the
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45 reference electrode and the working electrode, respectively.
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54 A $\text{CuCo}(\text{OH})_2$ precursor film was deposited at -1.0 V (vs. SCE) in potentiostatic mode
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56 (VERSA-STAT3, Princeton Applied Research) for 300 s. Afterward, the deposited film was
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5 rinsed with deionized water and then dried at room temperature. The deposited film was
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8 annealed in an air atmosphere at 300 °C with a ramping rate of 2 °C/min for 2 h.
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10 11 12 **Materials characterization**

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14 The structural and chemical properties of the annealed film were studied using high
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18 resolution X-ray diffraction (XRD) with Ni-filtered CuK α radiation [$k\alpha = 1.54056 \text{ \AA}$] (X_pert
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20 PRO, Philips, Eindhoven, Netherlands) and X-ray photoelectron spectroscopy (XPS, VG
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22 Multilab 2000, Thermo VG Scientific, UK) with a monochromatic Mg-K α (1253.6 eV) radiation
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24 source, respectively. The surface morphology, chemical composition, and crystallinity of the
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27 sample were examined using field emission scanning electron microscope (FE-SEM, Model:
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29 JSM-6701F, JEOL, Japan), energy-dispersive X-ray spectroscopy (EDS), transmission electron
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32 microscopy (TEM, JEOL 2010), and selected area electron diffraction (SAED), respectively.
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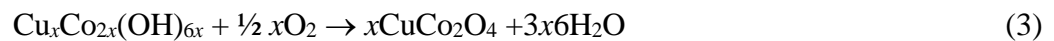
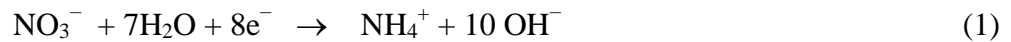
36 37 **Electrochemical Measurements**

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39 The CuCo₂O₄ film on NF was punched into circular plates with a diameter of 12 mm, and
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41 served directly as the working electrode. For the evaluation of the electrochemical properties of
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44 the CuCo₂O₄ nanosheet anode, a CR2032-type coin cell was assembled in an argon gas glove
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46 box (KOREA KIYON), in which, both H₂O and O₂ levels were kept below 0.1 ppm. Lithium
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48 metal foil was used as the counter electrode (cathode), a Celgard 2300 microporous membrane
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50 was used as the separator and 1M LiPF₆ dissolved in a mixture of ethylene carbonate and
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52 dimethyl carbonate (1:1 vol %) was used as the electrolyte solution. The electrochemical
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55 properties of the CuCo₂O₄ anode were studied using cyclic voltammetry (CV), galvanostatic
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5 charge-discharge, and electrochemical impedance spectroscopy (EIS) using a Bio-Logic (MPG-
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8 2) battery cycler.

13 **Results and discussion**

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15 A brownish bimetallic (Cu,Co) hydroxide precursor film was co-electrodeposited on the
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17 NF surface through the reactions of (Cu^{2+} , Co^{2+}) ions with OH^- , originated from the reduction of
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19 NO_3^- at the positive electrode. The generation of OH^- ions during the electrodeposition process
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21 were raised the local pH value, resulting in the uniform precipitation of mixed (Cu, Co)
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23 hydroxide on the NF surface. After this, (CuCo) hydroxide was thermally transformed into
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25 CuCo_2O_4 on the NF substrate. The corresponding electrochemical reactions are described as
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27 follows [15,16]:



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42 Fig. 1(a) shows the XRD pattern of the CuCo_2O_4 nanosheets on the NF substrate. It contains
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44 three strong peaks indicated by “#”, corresponds to the NF substrate. The as-deposited
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46 $\text{CuCo}(\text{OH})_2$ precursor film does not show any diffraction peaks, revealing its amorphous nature
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48 (Fig. S1). The clear diffraction peaks at 31.40° , 37.0° , 39.0° , 59.48° and 65.54° are observed and
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50 indexed to the (220), (311), (222), (511) and (440) planes of the spinel cubic CuCo_2O_4 phase,
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52 respectively (JCPDS card no. 01-1155). No other impurity peaks are observed.

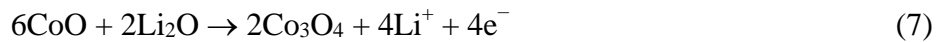
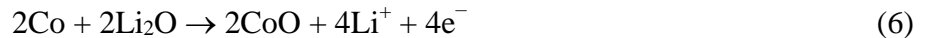
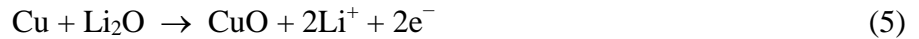
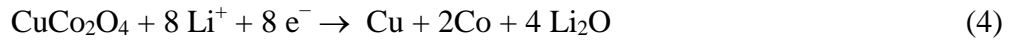
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55 The composition and oxidation states of the CuCo_2O_4 nanosheets were characterized using
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57 EDS and XPS measurements. The atomic ratio of Cu to Co in the annealed sample is ~ 1:2

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5 (Table S1). The survey XPS spectrum (Fig. S2) shows the presence of Cu, Co, and O with the
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7 carbon (for calibration). Fig. 1 (b-d) depicts the deconvoluted core-level spectra of Cu 2p, Co 2p,
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9 and O 1s, respectively. The core-level Cu 2p spectrum consists of two main peaks at binding
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11 energies of 933.5 and 953.4 eV. In addition, two shake-up satellite peaks (indicated by “Sat.”)
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13 are also observed at 942.1 and 962.1 eV (Fig. 1b), confirming the characteristic Cu^{2+} [17]. The
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18 Co 2p peak is deconvoluted into Co $2p_{3/2}$ and Co $2p_{1/2}$ spin-orbit doublets at 779.8 and 794.8 eV,
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20 respectively (Fig. 1c), together with a spin-energy separation of 15 eV, indicating the presence of
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22 mixed Co^{2+} and Co^{3+} . The presence of the two shake-up satellite peaks reveal that cobalt has a
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24 spinel structure [18]. The deconvoluted spectrum of O 1s in Fig 1d consists of three components.
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27 The O1 component at 529.4 eV corresponds to metal-oxygen bonds. The well-resolved O2
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29 component at 531.2 eV is associated with a larger number of defect sites with low oxygen
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31 coordination. The O3 component at 532.6 eV is assigned to the multiplicity of physi-sorbed and
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Fig. 2 (a) shows the FE-SEM image of the CuCo_2O_4 nanosheets. The uniform nanosheets are vertically formed on the NF substrate and interconnected to each other. The thickness of the nanosheets is about 10 -15 nm. This unique morphology is beneficial for ionic transport as well as electron transfer. The morphology of the as-deposited $\text{CuCo}(\text{OH})_2$ nanosheets does not change after annealing (Fig. S3). It is seen that the nanosheets are porous in nature (Fig. 2 (b)). From the HR-TEM analysis shown in Fig. 2(c), the spacing between adjacent planes is found to be ~ 0.14 nm which corresponds to the (440) plane of CuCo_2O_4 and is in agreement with the XRD data. The corresponding selected-area electron diffraction (SAED) pattern (Fig. 2d) exhibit well-defined diffraction rings with (111), (311) and (222) planes, revealing that the prepared material

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5 is spinel CuCo_2O_4 with a polycrystalline nature. Moreover, uniform distribution of the Cu and
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8 Co elements in the desired composition are observed in the film (Fig. 2 (e) and Table S1)

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10 Fig. 3a shows the cyclic voltammetry curves (CVs) for the CuCo_2O_4 nanosheet electrode
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12 for the 1st, 2nd, 3rd, and 4th cycles at a scan rate of 0.1 mV/s. In the cathodic scan of 1st cycle, two
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14 well-defined strong reduction peaks are observed at 1.0 and 0.7 V, which correspond to the
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16 reduction of Co^{3+} to Co^{2+} to metallic Co or Cu nanoparticles, respectively and formation of a
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18 solid electrolyte interface (SEI) layer[8,12]. In the anodic scan, two broad oxidation peaks are
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20 found at ~1.55 and ~2.10 V, which can be attributed to the oxidation of metallic Co and Cu into
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22 Co_3O_4 and CuO , respectively [5]. Similarly, the 2nd, 3rd and 4th CV curves indicate a good
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24 reversibility of the redox reaction. Because of the irreversible electrochemical reaction and the
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26 SEI layer formation after the 1st discharge cycle, the reduction peaks shift towards a higher
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28 potential side. On the contrary, the oxidation peaks are observed at similar potential values. The
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30 electrochemical reactions involved in this processes are as follows [8, 12]:
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47 Fig. 3 (b) shows the galvanostatic charge-discharge curves up to the 4th cycle at a constant
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49 current density of 0.1A/g. There are two discharge voltage plateaus at 1 and 0.7 V in the first
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51 curve whereas the charge voltage plateaus feature is observed at 1.55 and 2.10 V. These results
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53 are in good agreement with the potentials for the oxidation and reduction peaks in the 1st CV
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55 curve. The initial discharge and charge capacities are about 1244 and 1020 mAh/g, with a
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57 coulombic efficiency of 82 %. In the 2nd, 3rd and 4th cycles, the discharge potential plateaus shift
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5 towards a higher potential and the long discharge potential plateau changes to a sloping
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7 discharge curve, indicating that a stable SEI is formed during the 1st cycle [20].
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10 The rate capability of the electrode was measured with different current densities for each
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12 of five cycles and the results are shown in Fig. 3(c). As the current density increases up to 10 A/g,
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14 the discharge capacity decreases gently, thus showing an excellent high rate performance. As the
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16 current density recover to 1A/g, the discharge capacity (~1150 mAh/g) increases by ~ 12 %
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18 compared with the initial value (988 mAh/g) at the same current density. The increased capacity
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20 might have been associated with the phenomenon that the electrode material either partially loses
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22 its crystallinity or transforms to an amorphous-like structure during the cycling [21]. Fig. 3 (d)
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24 shows the cycling performance of the CuCo₂O₄ nanosheet electrode at a constant current density
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26 of 1 A/g for 200 cycles. The capacity of the electrode gradually decreases up to 90 cycles. After
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28 words, the capacity almost recovers to its initial value, presumably due to the activation of the
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30 electrode, the electrolyte decomposition or the formation of the SEI [22]. The CuCo₂O₄
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32 nanosheet electrode exhibits a capacity retention of 95% (1100 mAh/g) after 200 cycles.
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36 The EIS spectra were measured to understand the performance of the CuCo₂O₄ nanosheet
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38 electrode for before and after stability test. Fig. 4 shows the Nyquist plots obtained from the
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40 CuCo₂O₄ nanosheet electrode for fresh cell and after the stability test. Both the Nyquist plots
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42 exhibit a semicircles in the high-frequency region and a straight line in the low-frequency region.
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44 The straight line in the low-frequency region corresponds to the Warburg impedance (Z_w) and
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46 the semicircle are associated with the diffusion of Li⁺ ions into the electrode and the charge
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48 transfer resistance(R_{ct}), respectively[22]. Moreover, the solution resistance (R_s) as well as R_{ct}
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50 after the stability test is increased. Which can be attributed to the formation of a passivation layer
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52 due to the decomposition of the electrolyte [23].
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8 **Conclusions**

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10 A binder-free CuCo_2O_4 nanosheet anode for LIB has been successfully synthesized on
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12 NF substrate using an electrodeposition method followed by air annealing at 300 °C. The initial
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14 discharge specific capacity of the CuCo_2O_4 anode is 1244 mAh/g at 0.1 mA/g with a good
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16 coulombic efficiency of 82%. Also, it shows excellent high rate performance and stability with a
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18 capacity retention of 95% (1100 mAh/g) at a constant current density of 1 A/g after 200 cycles.
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21 Considering the facile synthesis as well as the excellent battery anode performance, the CuCo_2O_4
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23 nanosheet film in the present study has the potential to be an alternative to the current anode
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25 electrodes in LIB applications.
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35 **Acknowledgments**

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38 The authors would like to thank the financial support from the National Research Foundation
39
40 (NRF) of Korea (Grant nos. 2015M2A2A6A02045251, 2018R1A2B6007436,
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42 2016R1A6A1A03012877, and 2015R1D1A1A01060743).
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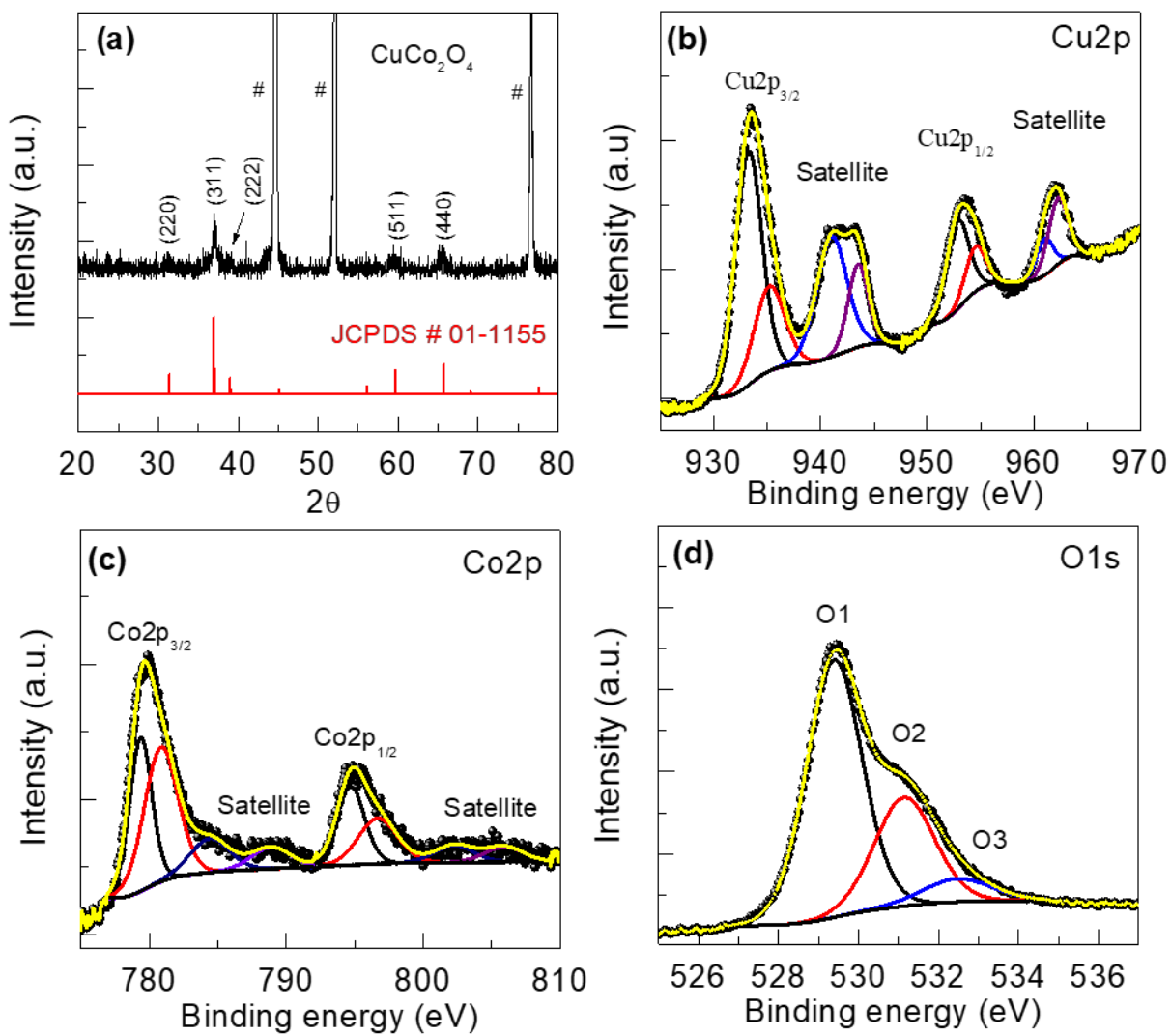


Fig. 1. (a) X-ray diffraction pattern, (b) Cu2p (c) Co2p and (d) O1s spectra of the CuCo₂O₄ nanosheet film.

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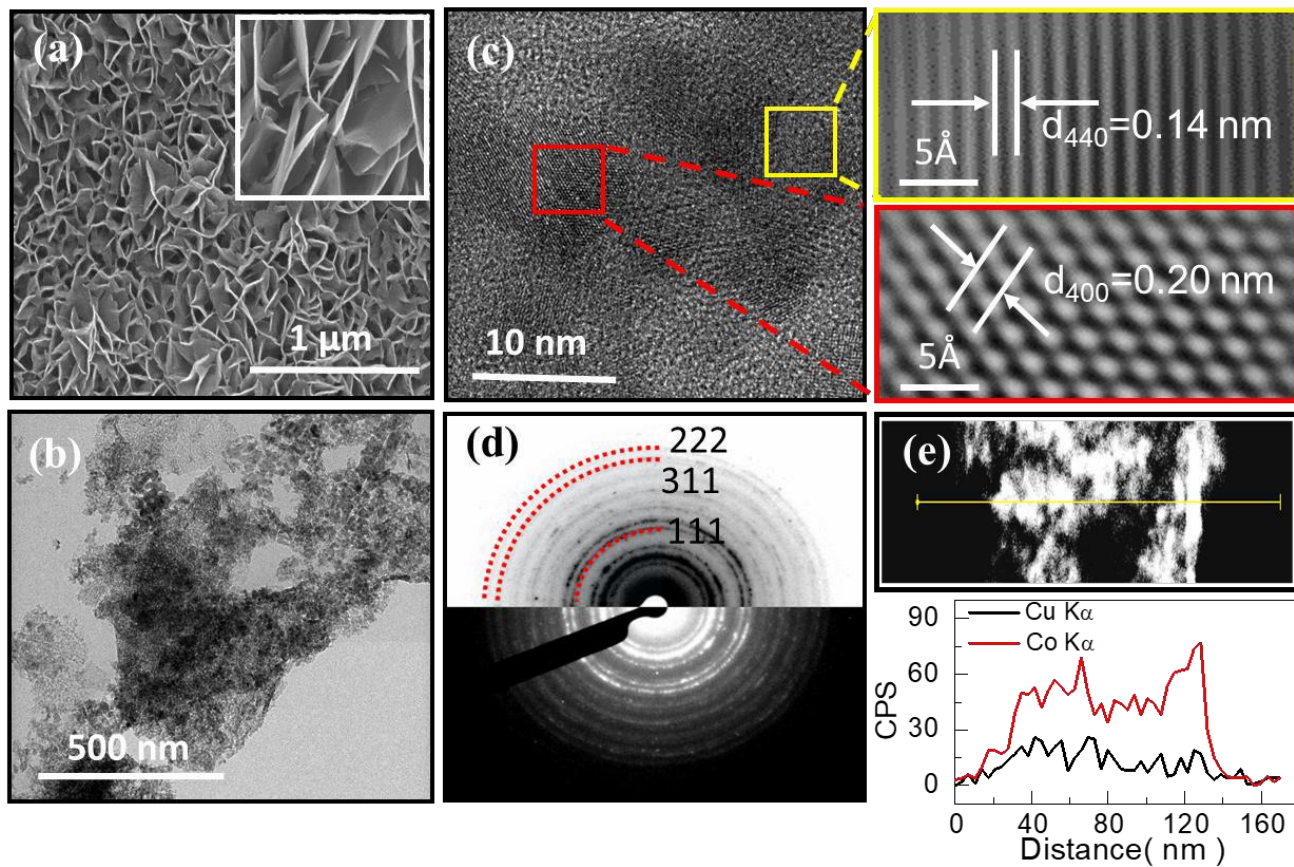


Fig. 2. (a) FE-SEM image (inset is high magnification), (b)TEM image, (c)HR-TEM image (inset is high magnification), (d) SAED pattern and (e) EDS elemental mapping with line scanning of the CuCo_2O_4 nanosheet film.

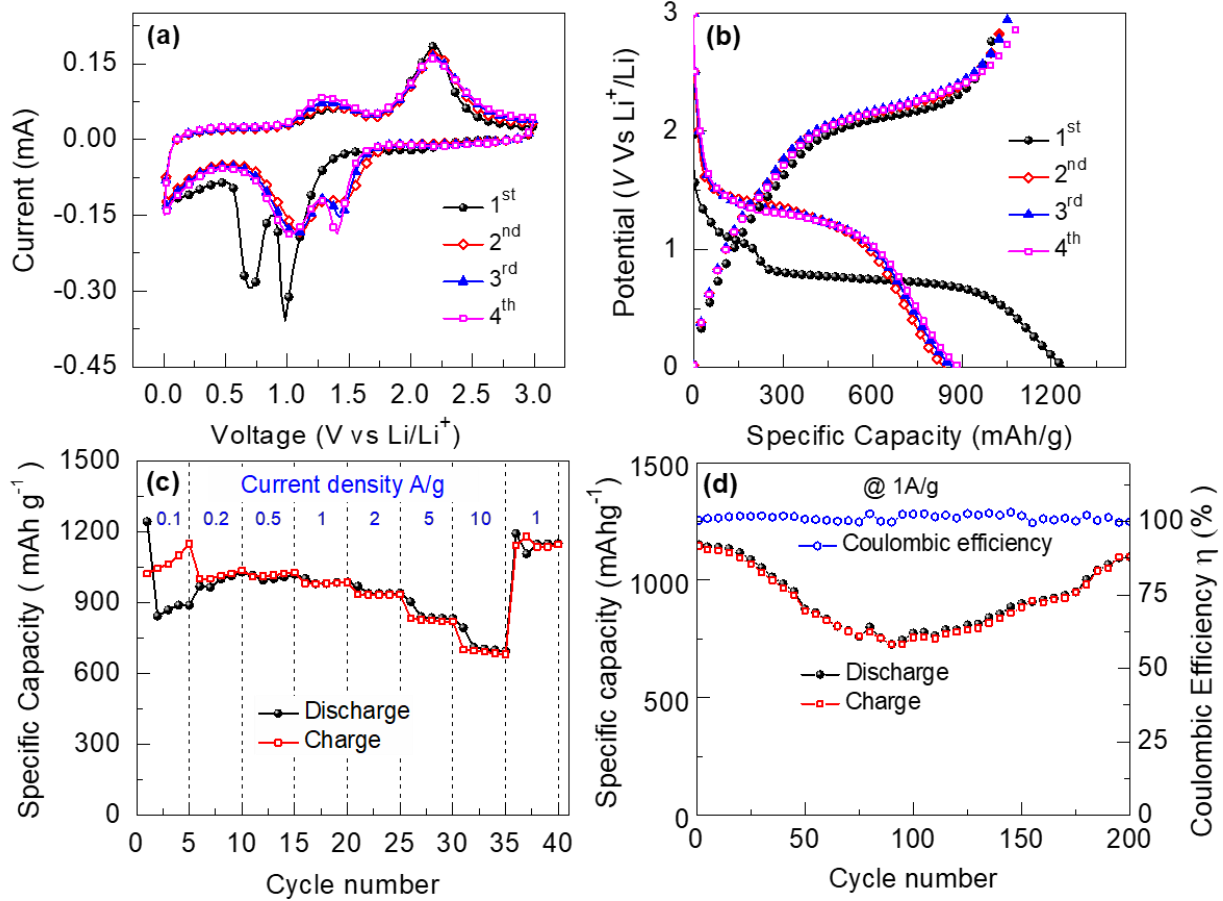


Fig. 3. (a) Cyclic voltammograms (CV) at a scan rate of 0.1 mV/s , (b) Galvanostatic charge/discharge curves at 0.1 A/g for first 4 cycles, (c) Rate capability measured at various current densities, and (d) cycling performance of the CuCo_2O_4 nanosheet electrode.

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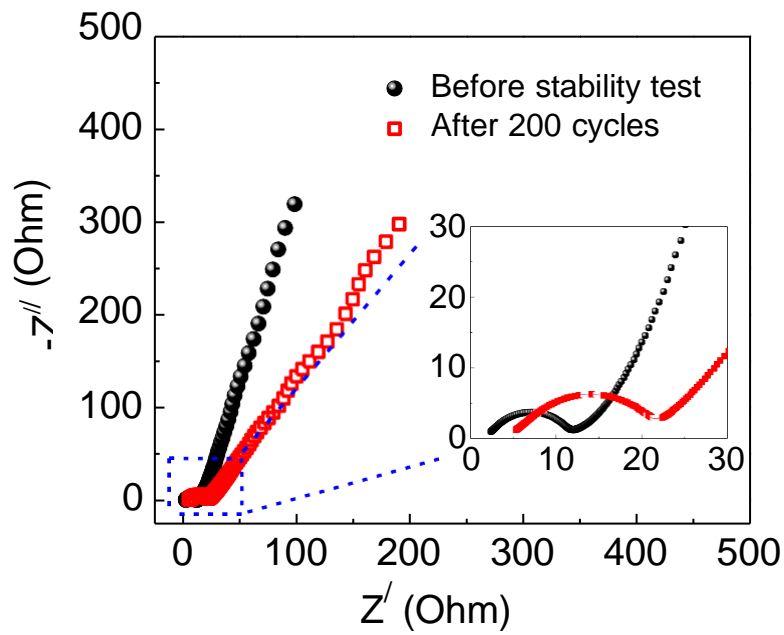


Fig. 4. Nyquist plots of the CuCo₂O₄ nanosheet electrode before and after 200 galvanostatic charge /discharge cycles.

Table 1: A LIB performance comparison between the various nanostructured CuCo₂O₄ - based anode materials

Morphology	Method	Specific capacity (mAhg⁻¹)	Current density (A g⁻¹)	Cycle number	Ref.
CuCo ₂ O ₄ nanoparticle	combustion	755	0.06	20	[5]
CuCo ₂ O ₄ nanoparticle/Graphene nanosheet	Solvothermal	1040	@0.1C	80	[6]
CuCo ₂ O ₄ hallow sphere	hydrothermal	930	0.1	150	[7]
3D CuCo ₂ O ₄ flower	Hydrothermal	1160	1	200	[8]
Carbon coated CuCo ₂ O ₄ concave polyhedron	Water bath	740	@0.1C	50	[9]
CuCo ₂ O ₄ /C nanofiber	electrospinning	865	0.2	400	[10]
CuCo ₂ O ₄ nanocubes /rGO	Microwave assisted solvothermal	570	1	350	[11]
CuCo ₂ O ₄ nanowall	hydrothermal	550	@0.375C	120	[12]
CuCo ₂ O ₄ nanosheet	electrodeposition	1100	1	200	This work

Supporting Information

Facile electrodeposition of high-density CuCo_2O_4 nanosheets as a high-performance Li-ion battery anode material

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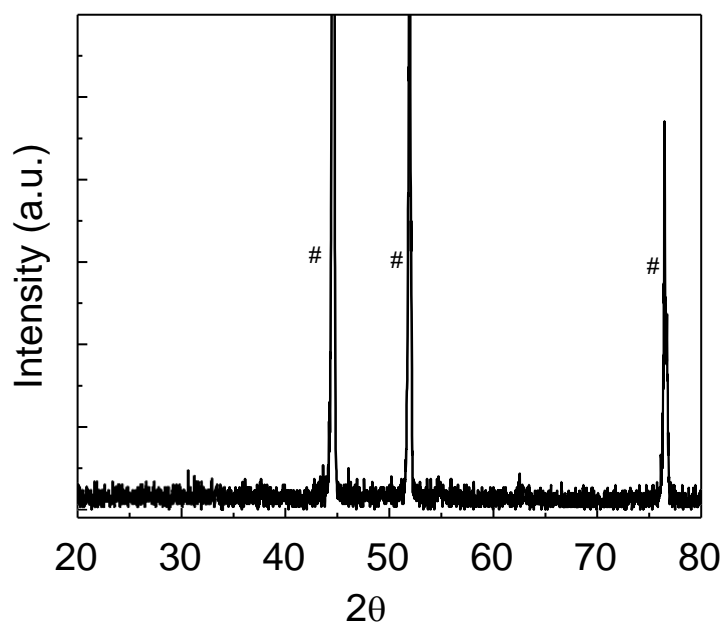


Fig.S1: X-ray diffraction pattern of as-deposited $\text{CuCo}(\text{OH})_2$ nanosheet electrode

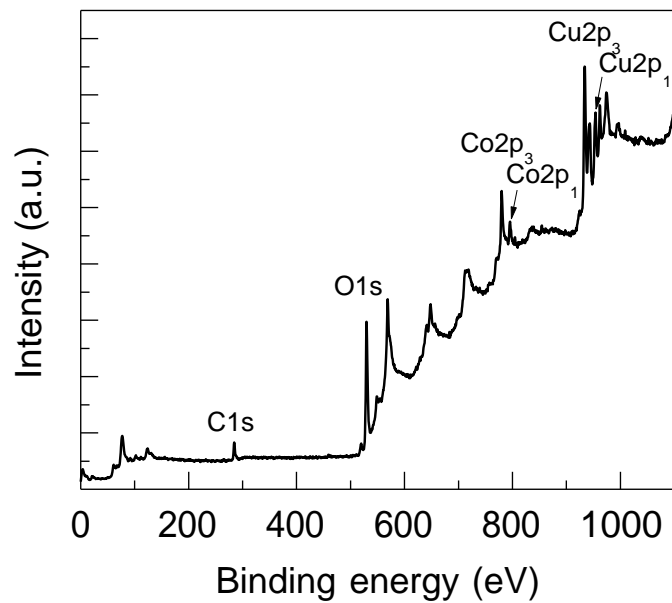


Fig. S2: XPS survey spectrum of CuCo_2O_4 nanosheet electrode.

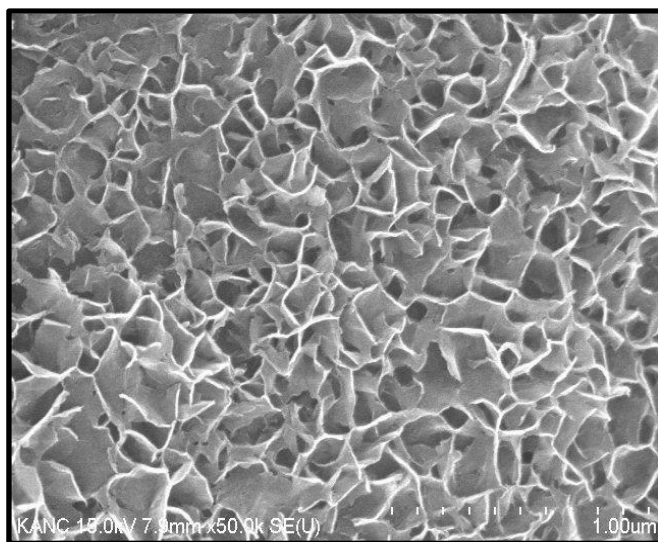


Fig. S3: As-deposited $\text{CuCo}(\text{OH})_2$ nanosheet films

Table S1. Compositional analysis of the CuCo_2O_4 nanosheets.

Elements	Cu	Co	O
At %	12.68	27.0	60.32