Electronic Supporting Information

Solvothermal Synthesis of Magnetic Copper Nitride Nanocubes with Highly Electrocatalytic Reduction Properties

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Experimental Details

Chemicals

All the reagents used in this work, including $Cu(NO_3)_2 \cdot 3H_2O$, ODA, ethanol, and cyclohexane were of analytical grade from the Beijing Chemical Factory of China and were used without further purification.

Characterization

The powder XRD patterns were recorded with a Bruker D8-advance X-ray powder diffractometer with CuK_α radiation (λ = 1.5406 Å). The specimen was prepared as follows. A small amount of product was dispersed in cyclohexane. Then, we dripped one drop of the resulting suspension onto a glass substrate. After it was dried at room temperature in air, we continued to drip another one until the final product fully covered the substrate. The 2θ range used in the measurement was from 10 to 80 in steps of 0.02 with a count time of 1 s. Transmission electron microscopy (TEM) images were recorded on Tecnai G2 F30 Field Emission Transmission Electron Microscope. The specimen was prepared as follows. A small amount of product was dispersed in cyclohexane. One drop of the resulting suspension was transferred onto a standard holey carbon-covered copper (or molybdenum) microgrid and dried at room temperature in air. X-ray photoelectron spectroscopy (XPS) measurements were carried out on a PHI-5702 multi-functional spectrometer using Al K_α radiation.

Synthesis of Cu_3N nanocubes

100 mg of $Cu(NO_3)_2 \cdot 6H_2O$ was added into 2.5 g of 1-octadecylamine (ODA) and 2 mL of oleylamine (OAm). The above mixture solution was degassed at 110 ^oC for 1 h under a flow of nitrogen, and under a blanket of nitrogen, quickly heated to 240 ^oC, and then kept at this temperature for 20 min. The black-brown mixture was cooled to room temperature. Ethanol (40 mL) was added to the mixture and the precipitate was collected by centrifugation at 8000 rpm. Finally, the product was redispersed in hexane.

Electrochemistry.

A glassy carbon (GC) disk electrode (3.0 mm diameter) was first polished with alumina slurries (0.05m) and then cleaned by sonication in 0.1 M HNO_3, H_2SO_4 and nano pure water successively for 10 min. The Cu_3N nanocubes dissolved in $CHCl_3$ were then dropcasted onto the clean GC electrode surface by a microliter syringe (the resulting electrodes were denoted as Cu_3N /GC). The particle films were dried by a gentle nitrogen stream for 2 min. Voltammetric

measurements were carried out with a CHI 660D electrochemical workstation. The Cu_3N /GC prepared above was used as working electrode. An saturated calomel electrode (SCE) reference electrode (in 3 M NaCl, aq.) and a Pt coil were used as the reference and counter electrodes, respectively. All electrode potentials in the present study were referred to the SCE reference electrode. Oxygen reductions were examined by first bubbling the electrolyte solution with ultrahigh purity oxygen for at least 15 min and then lanketing the solution with an oxygen atmosphere during the entire experimental procedure.

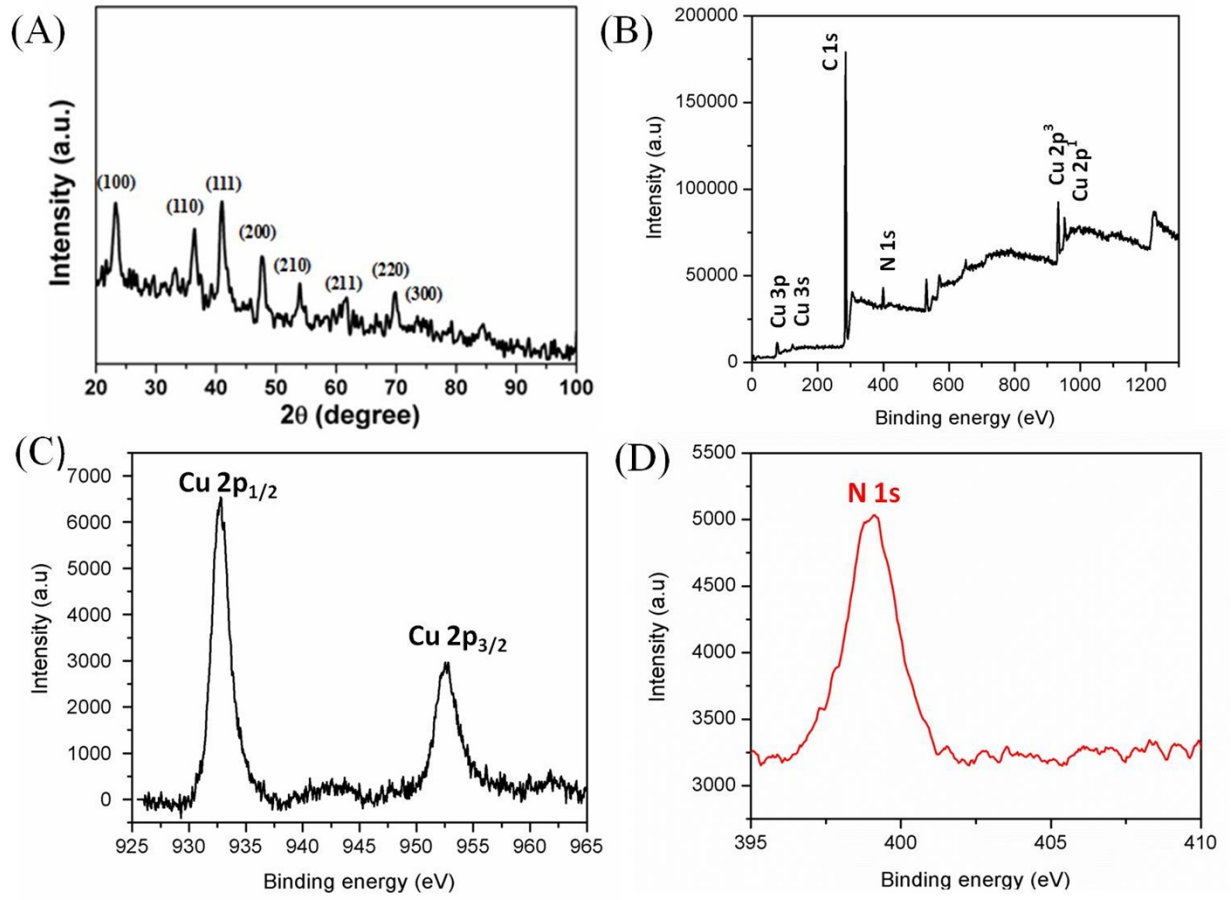

Fig. S1. (A) XRD pattern of Cu_3N nanocubes. (B) Survey spectrum of XPS pattern of Cu_3N nanocubes. (C) The Cu 2p spectrum of Cu (I). (D) The N 1s XPS spectrum.

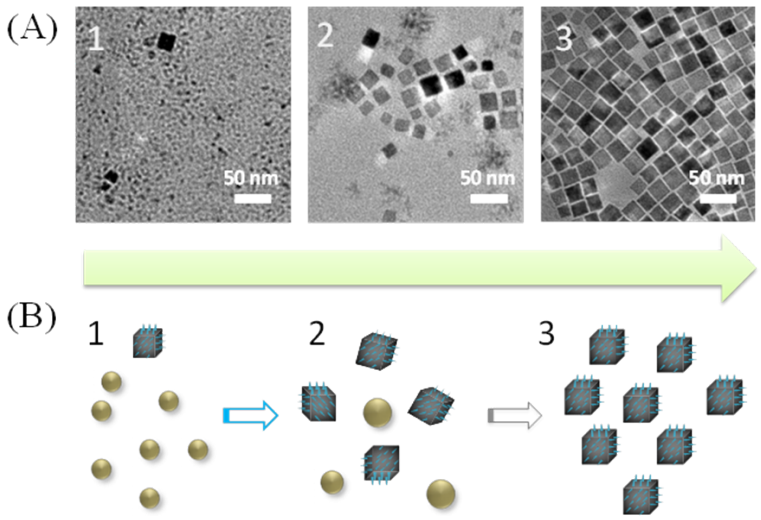

Fig. S2. (A) TEM of the Cu_3N at 240 °C with 2, 5 and 10 min. (B) The proposed growth procedure of Cu_3N nanocubes.

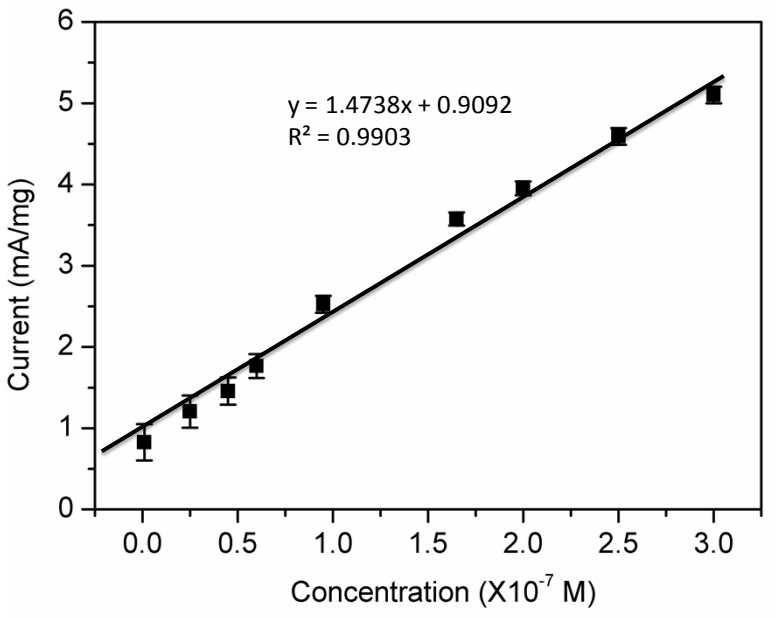

Fig. S3. The Linear relationship between peak current and NB concentrations.