

## Electronic Supplementary Information

### Three-dimensionalization of ultrathin nanosheets in a two-dimensional nano-reactor: macroporous CuO structures with enhanced cycling performance

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#### Chemicals

Copper acetylacetonate [Cu(acac)<sub>2</sub>] was purchased from Alfa Aesar. Dichloromethane was obtained from Richjoint. Sodium hydroxide (NaOH), Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>) and commercial CuO powder were purchased from Shanghai Co. All chemicals were used as received without further purification. All chemicals used in our experiment were of analytical grade and used without further purification. All solutions were prepared using ultrapure water.

#### Synthesis of macroporous CuO structures with ultrathin nanosheets

In a typical synthesis, 0.131 g of Cupric acetylacetonate [Cu(acac)<sub>2</sub>] was dissolved in 20 mL of dichloromethane and then the solution was transferred into a 50 mL test tube. After that, 20 mL of 3 mol/L NaOH aqueous solution was added into the tube to form a two-phase reaction system. After the tube was transferred into Electro-thermostatic Water Bath at 40 °C for 12 h, we can see the blue product on the inner surface turned into black gradually. After cooling to room

temperature, the black product at the inner surface of the tube was collected, washed with deionized water and ethanol several times and dried in the air at 40 °C.

### **Synthesis of CuO structures with thick blocks**

The 3D CuO structures with thick blocks(65 nm) were synthesized by reducing the concentration of NaOH aqueous solution to 1mol/L and increasing the reaction temperature to 60 °C. The black product was also collected, washed with deionized water and ethanol several times and dried in the air at 40 °C.

### **Characterizations**

The phase of the as-obtained samples was characterized by X-ray powder diffractometer (ULTIMA IV) with Cu K $\alpha$  radiation ( $\lambda=1.54056$  Å). Scanning electron microscopy (SEM) images of the as-obtained samples were obtained on a Hitachi S-4800 field-emission scanning electron microscope (FESEM). Transmission electron microscopy (TEM) images high-resolution electron microscopy (HRTEM) and the corresponding selected area electron diffraction (SAED) patterns were taken on the JEM-2100F transmission electron microscope at an acceleration voltage of 200 kV. The N<sub>2</sub> adsorption–desorption isotherms were measured using an ASAP-2000 surface area analyzer.

### **Photocatalytic measurements**

The evaluation of the photocatalytic activity of the as-prepared samples was performed by suspending 50 mg of CuO sample into a mixture of 50 mL of 10 mg/L Rhodamine B aqueous solution and 1 mL of hydrogen peroxide solution (H<sub>2</sub>O<sub>2</sub>, 30 wt%) in a beaker at ambient temperature. Before irradiation, the mixed solution was magnetically stirred in dark for 1 h to ensure establishment of an adsorption–desorption equilibrium of dye on the sample surface. Then, the photocatalytic test was conducted by irradiation with a daylight lamp (5 W). After irradiation for different times, the solution samples (3 mL) were taken out and dilute by 6 ml H<sub>2</sub>O then centrifuged for the absorbance measurements by the spectrophotometer.

### **Electrochemical measurements**

The electrochemical test was carried out in a CR2032-type coin cell. The working electrodes were prepared by mixing 80 wt% active materials (the obtained CuO micro-flowers or commercial CuO), 10 wt% acetylene black and 10 wt% polyvinylidene fluoride (PVDF) dissolved in N-methylpyrrolidinone (NMP). The formed slurry was coated onto a copper foil. After solvent evaporation at room temperature and drying in a vacuum oven at 80 °C for 12 h, the electrodes were assembled into coin cells with a Celgard separator membrane and lithium metal as the counter and reference electrode. The electrolyte solution was 1 mol/L LiPF<sub>6</sub> in ethylene carbonate (EC)–diethyl carbonate (DEC) (1:1 by volume). The test cells were assembled in an argon-filled (99.999%) glovebox. The galvanostatic charge–discharge and cycling performance measurements were carried out using a LAND CT2001A Battery Testing System (Wuhan Land Electronic Co. Ltd., China) between 0.05 V and 3.0 V (vs. Li/Li<sup>+</sup>). The data of first cycle in Fig. 4 was recorded after a pre-running to ensure adequate contact between CuO electrodes and electrolyte.

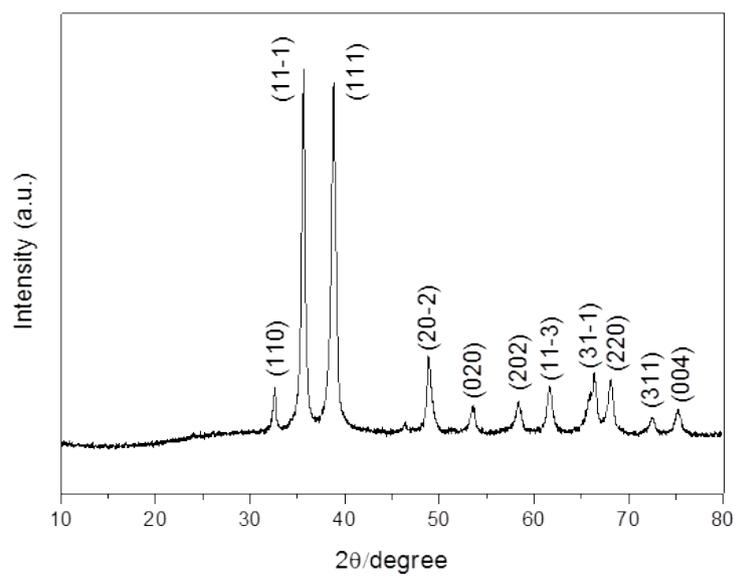


Fig. S1 XRD pattern of the as-prepared 3D macroporous CuO structures composed by ultrathin nanosheets.

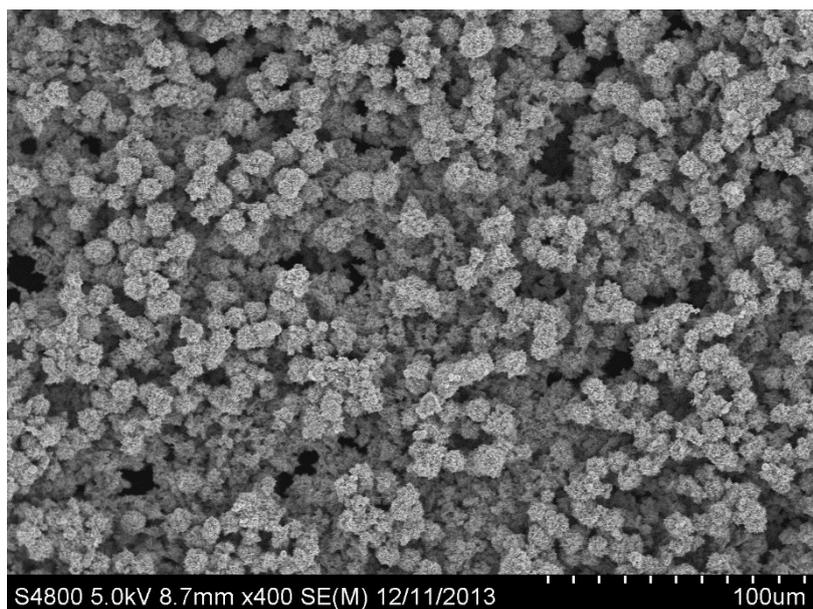


Fig. S2 the low-magnified FESEM image of the as-prepared 3D macroporous CuO structures composed by ultrathin nanosheets.

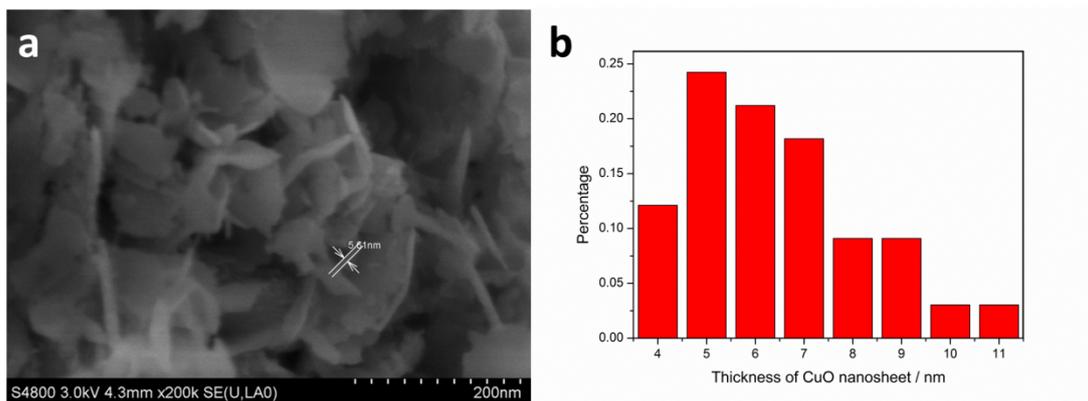


Fig. S3 (a) High magnified SEM image taken from a part of the 3D macroporous CuO structures. (b) Thickness distribution histogram of CuO nanosheets

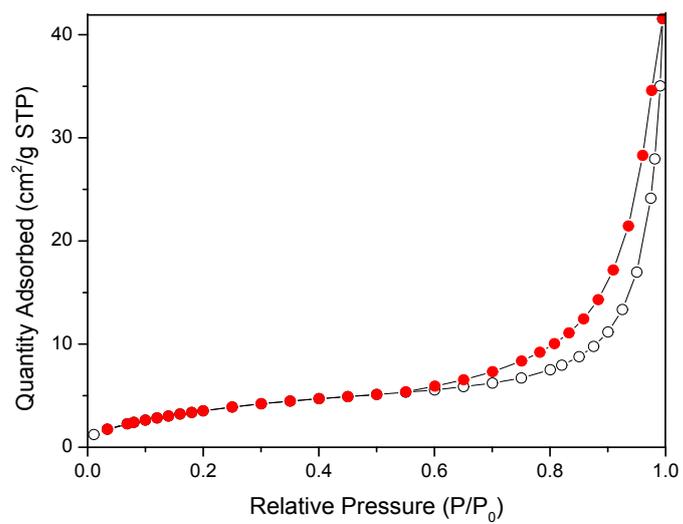


Fig. S4 Nitrogen adsorption-desorption isotherm of the macroporous CuO structures obtained at 77 k.

Table. S1 Thickness of CuO nanosheets in previous reports.

Thickness of CuO nanosheets	Reference
10-20 nm	17a, 17b
20-40 nm	17c, 17d
40 nm or more	17e, 17f, 17g

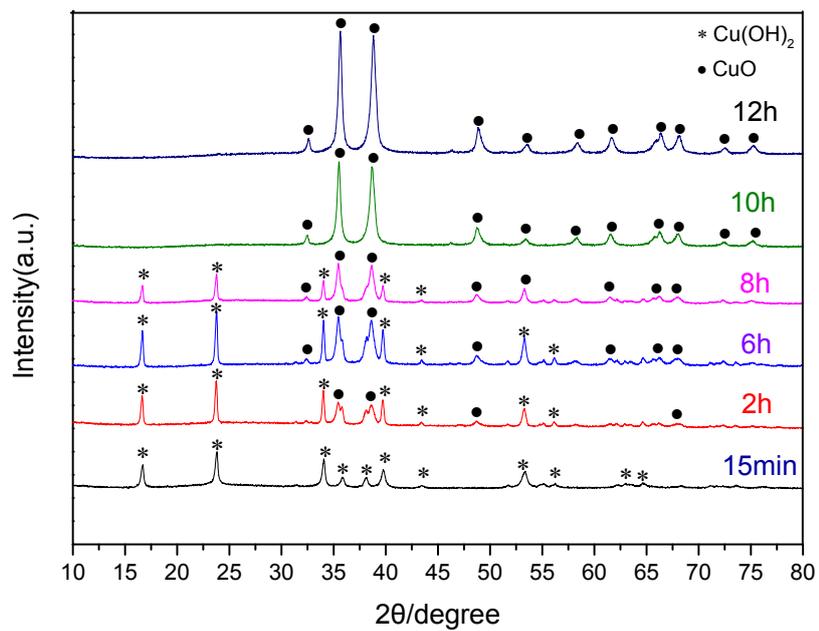


Fig. S5 XRD pattern of the synthesized samples after different duration.

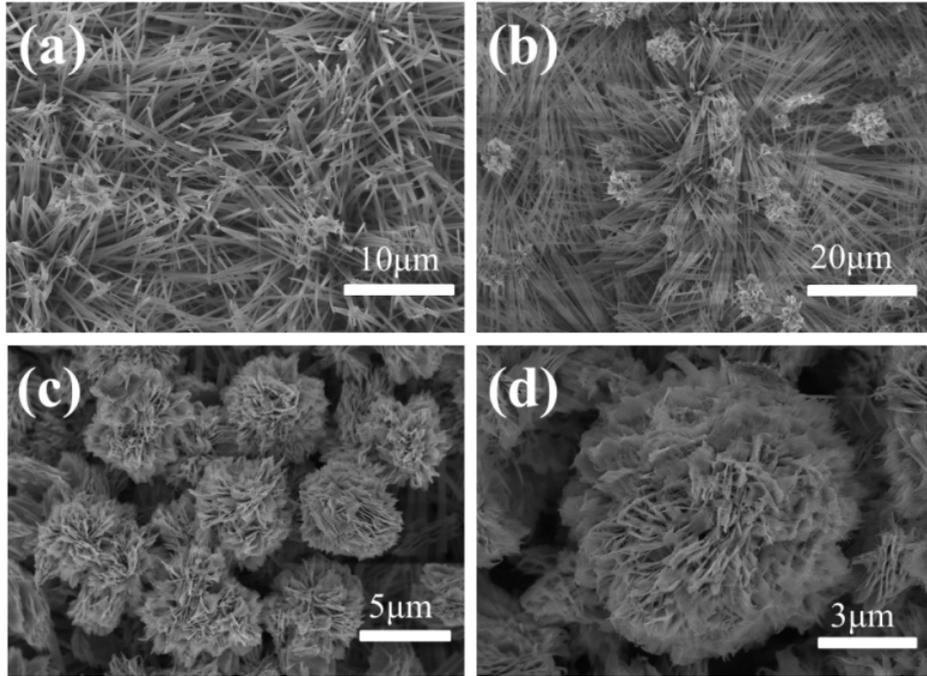


Fig. S6 SEM pictures of the synthesized samples at different time.(a) SEM image of the prepared  $\text{Cu}(\text{OH})_2$  nano-rods at 15 min; (b) SEM image of the product at 2h; (c) SEM image of the product at 6 h; (d) SEM image of the as-prepared  $\text{CuO}$  structures at 12 h.

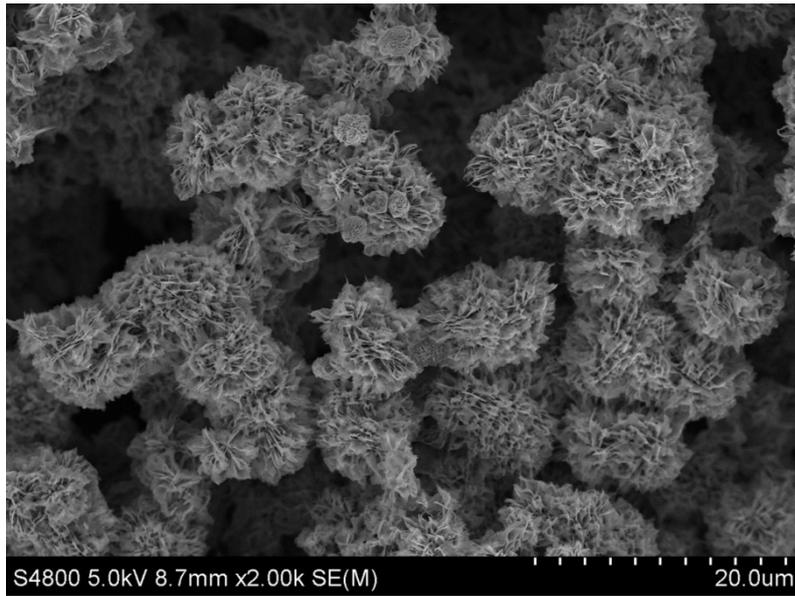


Fig. S7 SEM picture of 3D macroporous CuO structures composed by ultrathin nanosheets after five recycling, kept the structure well.

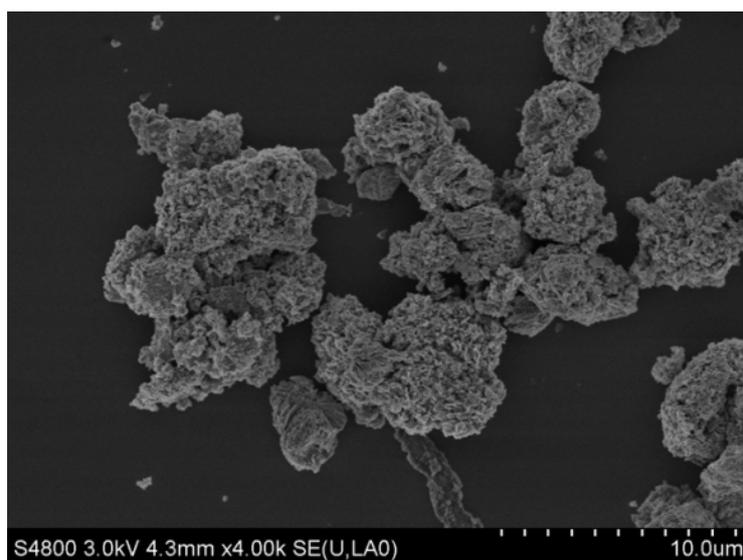


Fig. S8 SEM picture of CuO non-organized nanosheet after five recycling, serious reunion

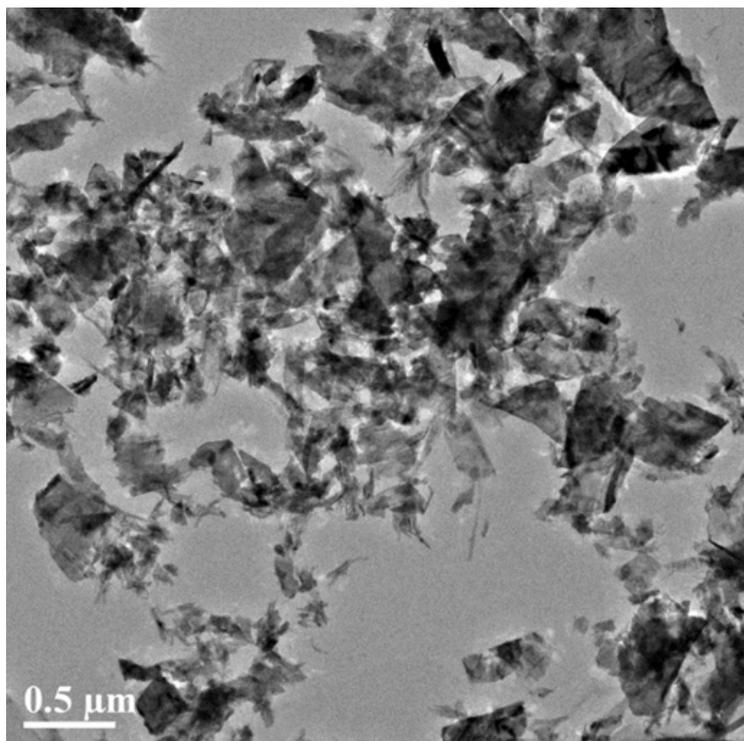


Fig. S9 TEM picture of non-organized CuO nanosheets

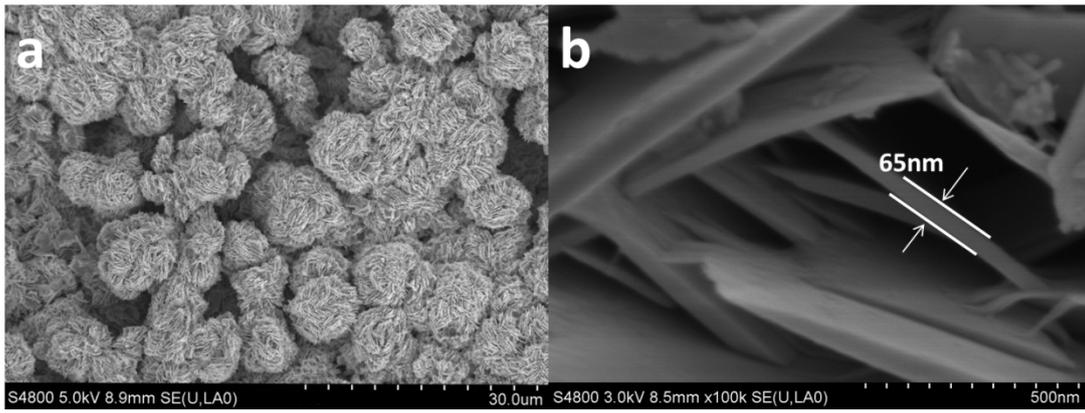


Fig. S10 Synthesized 3D CuO structures composed by thick blocks. (a) The overall appearance. (b) Thickness of the composed nanosheets.

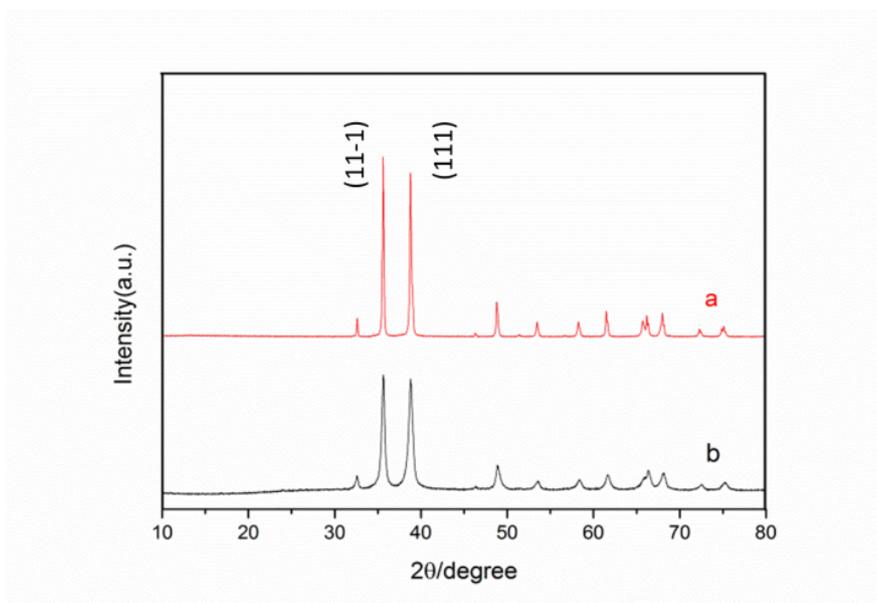


Fig. S11 XRD patterns of (a) commercial CuO and (b) synthesized 3D CuO structure composed by ultrathin nanosheets.