Electronic Supplementary Material (ESI) for RSC Advances.

Supplementary Information

A Facile One-Step Fabrication of a Novel Cu/MoS_2 Nano-Assembled Structure for Enhanced Hydrogen Evolution Reaction Performance

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Preparation of Cu/MoS_2 nano-assembled structure

Sodium molybdate (242 mg), thiourea (286 mg), and $CuCl_2 \cdot 2H_2O$ (2.18 mg) were added to 60 ml of a mixture solution of isopropyl alcohol and ethylene glycol (EG) (v/v = 1:4) and sonicated for 30 min. A 1 M NaOH/EG solution was added to the mixture until a pH of 12 was reached, and then argon was blown into the mixture for 20 min. The mixture was microwaved for 120 s at a dynamic power of 1700 W by a microwave oven (Panasonic NE-1753) in a draught cupboard and then cooled naturally. Next, 1 M dilute nitric acid was added until a pH of 2 was reached. The product was collected by vacuum filtration and vacuum-dried at 60°C.

Preparation of MoS_2 nanosheets (MoS_2 NSs)

A liquid exfoliation technique of ultrasound probe sonication was used to obtain MoS_2 NSs. This method is less susceptible to the surrounding environment, simple operation and suitable for large-scale production. To begin with, 1 g of powder of MoS_2 was dissolved in 100 mL N-Methylpyrrolidone (NMP), which was placed in a glass vial for a 3.5 h ultrasound, under the ultrasound power of 300 W, maintaining the temperature at 20 °C. Then, the mixture was transferred to centrifuge tubes for the first centrifugal, which was at the speed of 1500 rpm for 60 min and under the temperature at 10 °C. The top two-thirds of the supernatant liquid were reserved, and ethanol was added thereto to 300 ml for dilution. After that, the dilution was added into an ultrasonic processor, sonicated with an ultrasound probe for 10 min to obtain the up two-thirds of the solution, and then the solution was centrifuged at 2000 rpm for 60 min, the supernatant was small-sized MoS_2 NSs.

Preparation of Cu-modified MoS_2 nanosheets (Cu/MoS_2 NSs)

MoS_2 NSs (20 mg), and $CuCl_2 \cdot 2H_2O$ (2.18 mg) were added to 60 ml of a mixture solution of isopropyl alcohol and ethylene glycol (EG) (v/v = 1:4) and sonicated for 30 min. A 1 M NaOH/EG solution was added to the mixture until a pH of 12 was reached, and then argon was blown into the mixture for 20 min. The mixture was microwaved for 120 s at a dynamic power of 1700 W by a microwave oven (Panasonic NE-1753) in a draught cupboard and then cooled naturally. Next, 1 M dilute nitric acid was added until a pH of 2 was reached. The product was collected by vacuum filtration and vacuum-dried at 60°C.

Material characterization and electrochemical evaluation

The morphologies of the electrocatalysts were observed by transmission electron microscopy (TEM) using a microscope operated at an accelerating voltage of 300 keV. XPS was used to record the elemental composition and the electron binding energy using a K-Alpha instrument. The HER activity of electrocatalysts was examined using linear sweep voltammetry (LSV) with a scan rate of 5 mV s^{-1} in 0.5 M H_2SO_4. The LSV was carried out using a CHI660D electrochemical workstation in a standard three-electrode setup with a saturated calomel electrode (SCE) as the reference electrode and a Pt foil as the counter electrode. Prior to the electrochemical measurements, the electrolyte was degassed by bubbling argon gas through it for 1 h; stable polarization curves were recorded after 20 cycles.

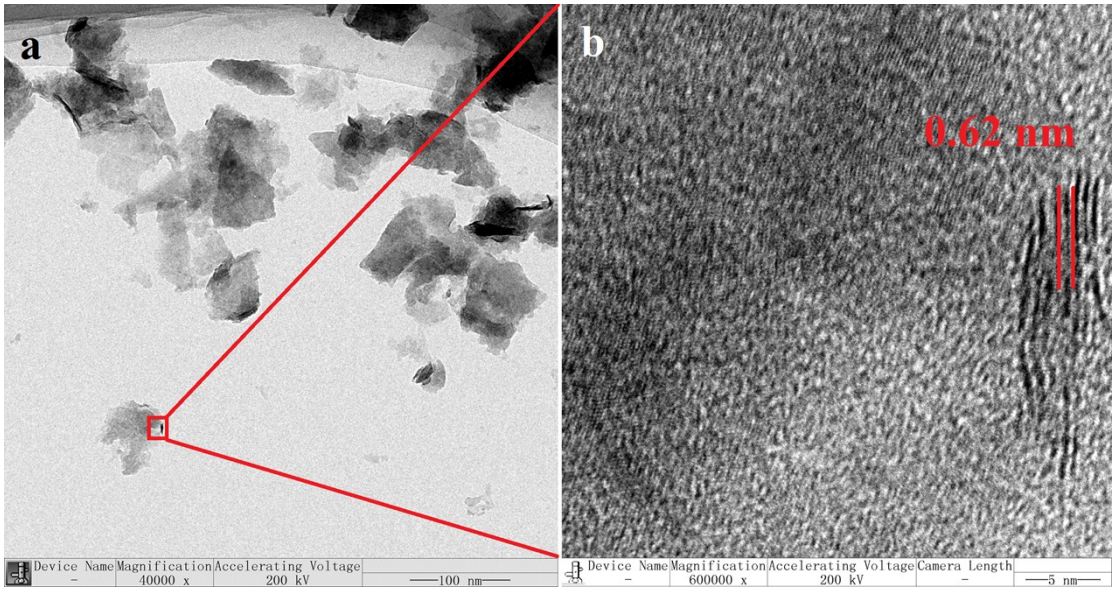

Fig. S1 TEM image (a) and HRTEM image (b) of MoS_2 NSs. The images indicated that the active edges of the lager-sized MoS_2 NSs were finite.

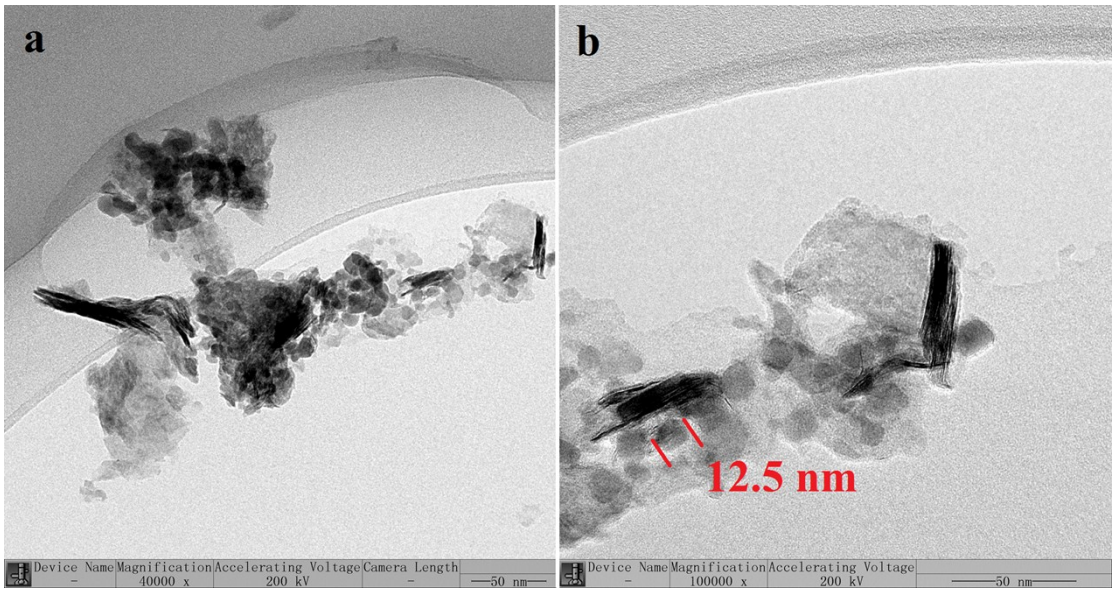

Fig. S2 TEM image (a) and HRTEM image (b) of Cu/MoS_2 NSs nanohybrid. HRTEM indicated that having many highly crystalline Cu NPs covering the edges of the MoS_2 NSs may lead to the reduction of HER electrocatalytic performance.

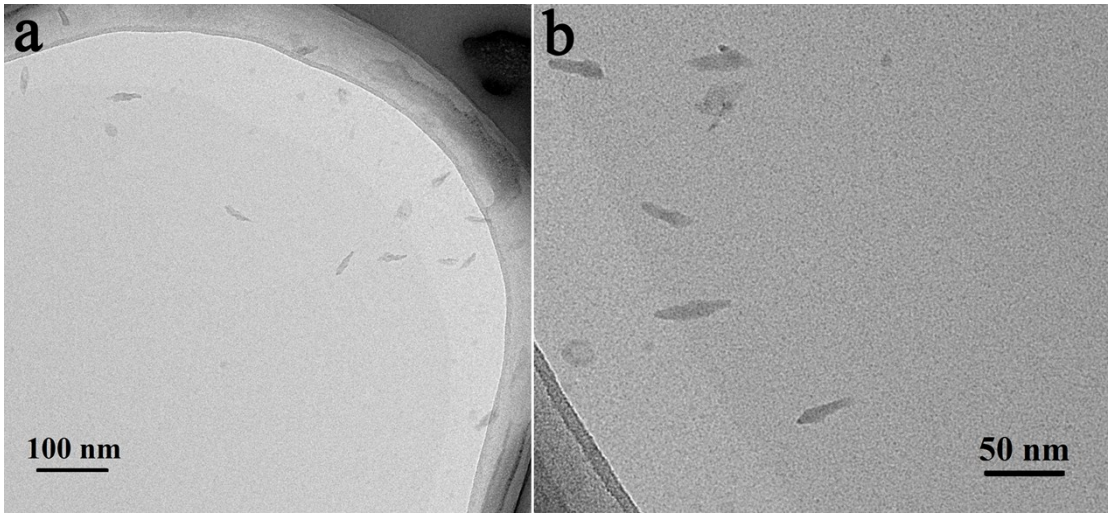

Fig. S3 TEM images of small-sized MoS_2 NSs.

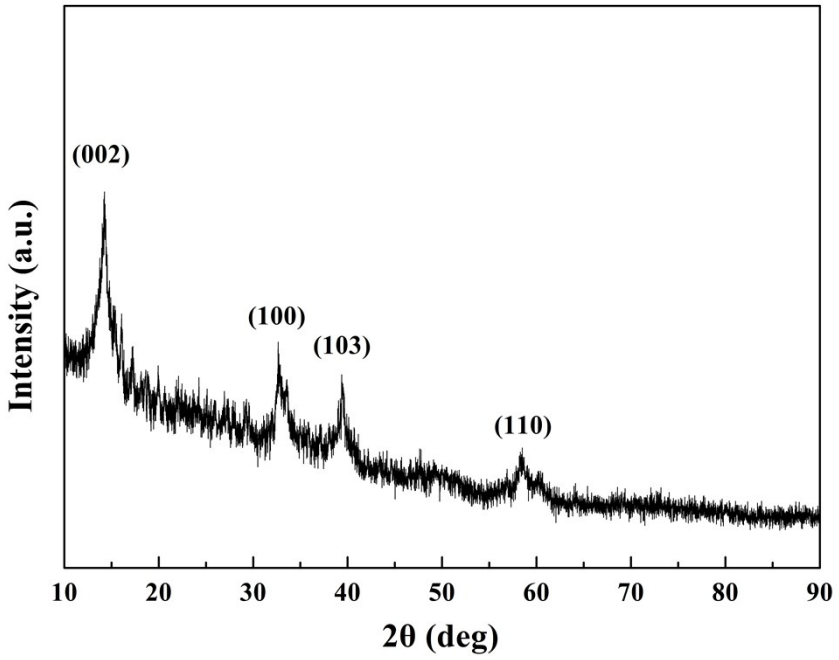

Fig. S4 XRD pattern of small-sized MoS_2 NSs.