Supporting Information

Alloying Au into Cu/Cu_2O/Nickel Foam Photoanode for Solar-Enhanced Hydrogen Production Coupled with Glucose Oxidation

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1. Chemicals and Experimental Session

1.1. Chemicals

Copper (II) nitrate trihydrate ($Cu(NO_3)_2.3H_2O$, ≥99.5 %), gold(III) chloride trihydrate ($HAuCl_4.3H_2O$, ≥99.9 %), glycerol ($C_3H_8O_3$, ≥99 %) were supplied from Merck. Glucose ($C_6H_{12}O_6$, 99.9 %) and potassium hydroxide (KOH, 99 %) were purchased from Sigma Aldrich and used without further purification. Nickel foam (NF) (99.9 %) was used as support and provided by Beijing Beike 2D Materials Co., Ltd. Deionized water was used throughout all experiments.

1.2. Synthesis of Au-Cu/NF and compared samples

1.2.1 Synthesis of Au-Cu/NF

The Au-Cu/NF sample was fabricated via a facile hydrothermal method. Briefly, 1.03 g $Cu(NO_3)_2.3H_2O$, 0.039 g $HAuCl_4.3H_2O$, and 7.30 g $C_3H_8O_3$ were dissolved in 50.0 mL deionized water and stirred continuously for 1.5 hours. The resulting solution was transferred into a Teflon-lined autoclave. Then, the NF (3 cm x 3 cm), pre-treated in 50.0 mL of 1.0 M HCl solution for 45 minutes in a sonication bath, followed by sequentially washing with ethanol and deionized water, was placed in the prepared mixture before being hydrothermally treated at 160 ^oC for 16 h. After cooling to room temperature, the obtained material (denoted as Au-Cu/NF) was subsequently washed with deionized water and ethanol to remove residual reactants, and then dried at 60 °C for further experiments.

1.2.2 Synthesis of Cu/NF

A similar synthetic procedure was utilized for the Cu/NF sample without the addition of Au precursor.

1.3. Characterizations

The morphology and chemical analysis of as-prepared catalysts were examined by scanning electron microscopy (SEM, Tescan Vega) equipped with an energy-dispersive X-ray spectroscopy (EDS) detector (Bruker 630M). High-resolution transmission electron microscopy (HR-TEM) of samples was conducted by the JEOL system. The powder X-ray diffraction (XRD) patterns were collected by XRD, Bruker D8. Raman spectra were recorded with a LabRAM spectrometer (Horiba). X-ray photoelectron spectroscopy (XPS) measurements were carried out with a Thermo Fisher Scientific spectrometer. The UV-Vis absorption spectra were obtained by a UV-Vis absorption spectrophotometer (Cary 300) in the wavelength range of 200 – 800 nm.

1.4. Photoelectrochemical measurements

The photoelectrochemical performance of the samples was conducted by an electrochemical workstation (PGSTAT204, Metrohm) in a standard three-electrode configuration, in which as-prepared samples (with an exposed area of 1×1 cm^2), platinum, and Ag/AgCl served as working, counter, and reference electrodes, respectively. The light source was supplied by a 150 W Xenon lamp (Sciencetech). The solutions of 1.0 M KOH and 1.0 M KOH + 0.10 M glucose were employed as the electrolytes. The measured potentials were converted to potential versus the reversible hydrogen electrode (vs. RHE) via the following equation:

$$E_{RHE} = E_{Ag/AgCl} + 0.059 \times pH + E^0_{Ag/AgCl} \quad (1)$$

To activate the material, cyclic voltammetry (CV) was first performed at a scan rate of 50 $mV \cdot s^{-1}$ for 10 cycles (**Figure S1**). Linear sweep voltammetry (LSV) was conducted at a scan rate of 5 $mV \cdot s^{-1}$. Electrochemical impedance spectroscopy (EIS) was carried out at a potential of 1.32 V vs. RHE over a frequency range of 0.1 Hz to 100 kHz with an amplitude of 10 mV. The stability of the catalyst was evaluated via a chronoamperometry test in an H-cell at a potential of 1.62 V vs.

RHE for 15 consecutive cycles, corresponding to a duration of 45 hours. The amount of evolved hydrogen was measured by the water displacement method.

2. Results

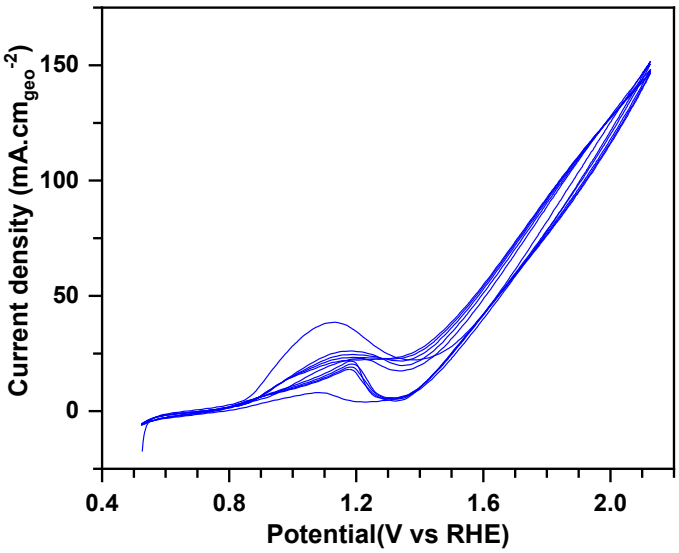

Figure S1. The CV curves of Au-Cu/NF sample electrode recorded for 10 cycles in 1.0 M KOH + 0.1 M glucose solution

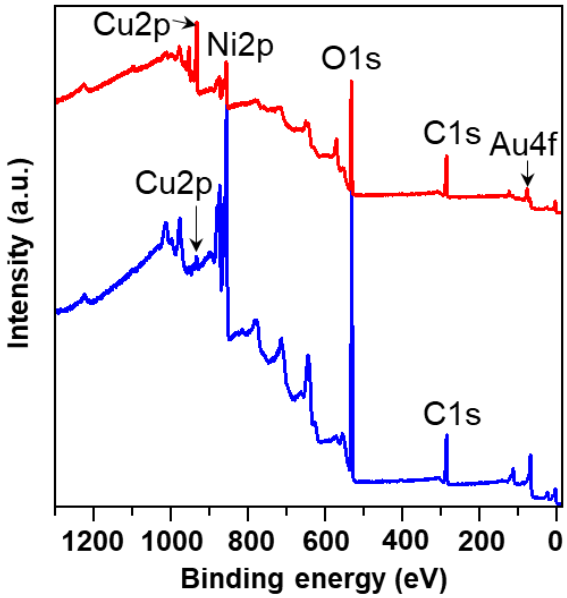

Figure S2. The survey XPS spectrum of Au-Cu/NF and Cu/NF.

Table S1. Elemental composition of as-synthesized samples according to XPS survey spectra

Name	% Atom	
	Au-Cu/NF	Cu/NF
C1s	9.83	28.6
Cu2p	28.56	0.89
Ni2p	37.36	19.36
O1s	24.14	51.16
Au4f	0.11	

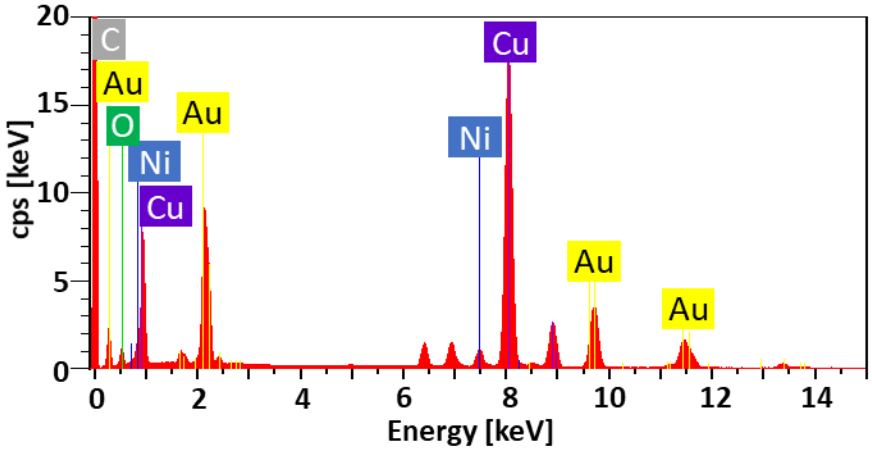

Figure S3. EDS spectrum of Au-Cu/NF sample.

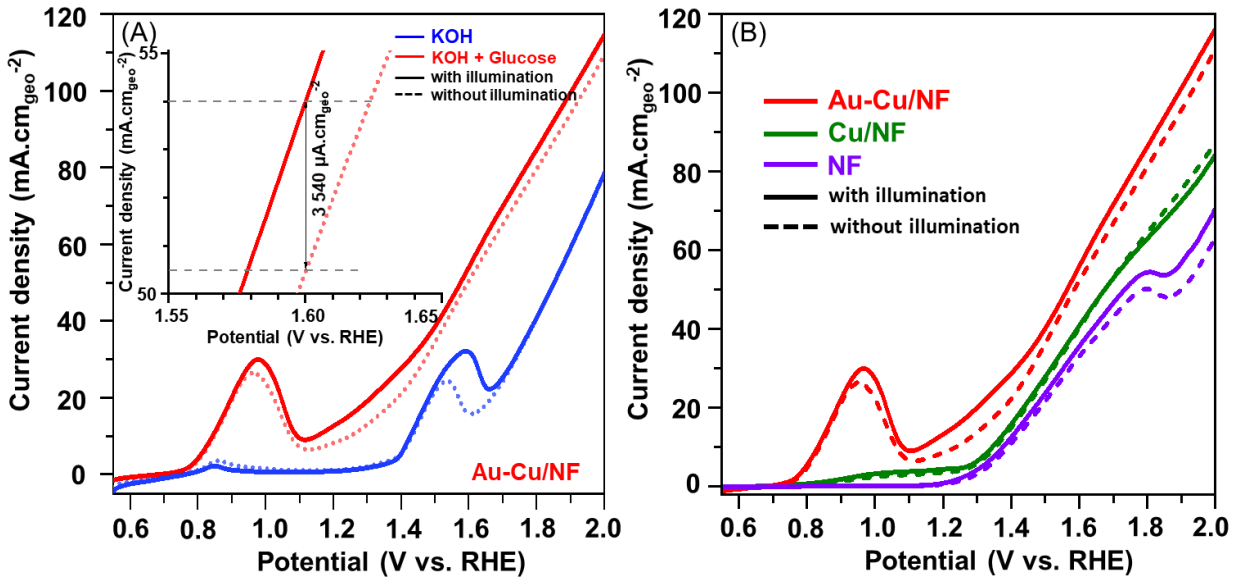

Figure S4. LSV-normalized curves of Au-Cu/NF sample with and without glucose (A) and LSV-normalized curves (B) of as-prepared Au-Cu/NF, Cu/NF, NF.

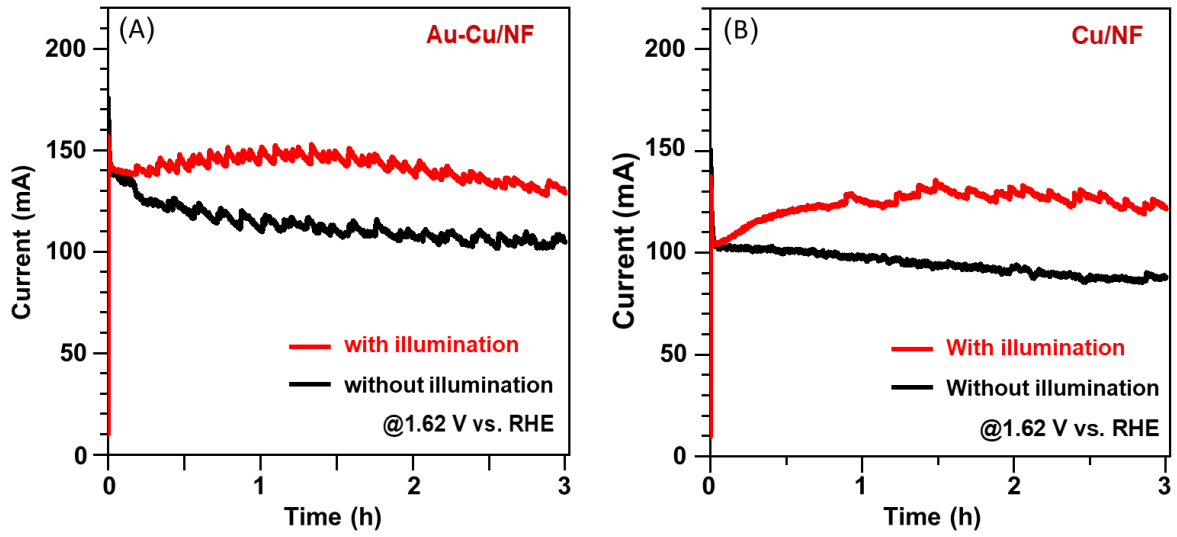

Figure S5. The chronoamperometry curves of as-prepared sample in 1.0 M KOH + 0.10 M Glucose.