Introduction

CO₂ is a rich C₁ resource and its conversion into chemicals has attracted more and more attention.¹⁻³ Particularly, reducing CO₂ into CO, which is a key starting material for the synthesis of important chemicals and commodities, is of great importance.⁴ Among the transformation approaches, the electrochemical CO₂ reduction reaction (CO₂ RR) into CO using an electrocatalyst is a promising way.⁵⁻⁹ Compared with precious metal electrocatalysts (Au, Ag and Pd),¹⁰⁻¹² it is more desirable to develop alternative earth-abundant metal catalysts for the CO₂ RR to CO. However, non-noble metal electrocatalysts often suffer from low activities and selectivities. It has been proposed that the formation of metal alloy or bimetallic catalysts (especially with noble metals) can improve the efficiency of non-preciuous metals for the CO₂ RR, through tuning their binding energy to reaction intermediates and regulating their inherent activity.¹³⁻¹⁵ However, the performances of these electrocatalysts for the CO₂ RR still need improvement in terms of two issues. First, other carbon products (e.g. formic acid, methane, etc.) are inevitably produced in many catalytic systems. Second, the current density is usually low, especially at relatively low overpotentials. It is challenging to develop non-precious metal electrocatalysts with combined high activity, selectivity and current density for the CO₂ RR into CO.

Here we demonstrate for the first time the formation of Cu/Ni alloy nanoparticles embedded in a three-dimensional (3D) nitrogen–carbon network. Outstandingly, the as-synthesized catalyst can effectively convert CO₂ into CO with a maximum faradaic efficiency of 94.5% and current density of 18.8 mA cm⁻² at a low applied potential of −0.60 V (versus reversible hydrogen electrode, RHE). Moreover, the catalyst is very stable during long-term electrolysis owing to the stabilization of the nitrogen–carbon network.

Results and discussion

To get the target product, a Cu/Ni bimetallic complex was first prepared by a hydrothermal method using copper powder (Fig. S1†) and nickel(II) nitrate hexahydrate as precursors. From scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images, the Cu/Ni bimetallic complex has a 3D network structure (Fig. 1a, d and S2†). After calcination of the Cu/Ni bimetallic complex at 900 °C for 1 h, the target product was obtained. It well maintains the network structure of the bimetallic complex, but it is embedded with nanoparticles in the range of 50–300 nm (Fig. 1b and c). The high-resolution transmission electron microscopy (HRTEM) image (Fig. 1e) shows a lattice fringe corresponding to the crystal plane...
(111) \((d = 0.21 \text{ nm})\) of the face centered cubic Cu/Ni bimetallic alloy,\(^{22,28}\) which is consistent with the X-ray diffraction (XRD) pattern (Fig. S3†). Energy dispersive X-ray spectroscopy (EDS) line scans on nanoparticles reveal that Cu and Ni elements are distributed nearly at the same locations (Fig. 1f), further proving the formation of the Cu/ Ni alloy. Energy dispersive X-ray elemental (EDX) mapping images show that C, N and O elements are evenly distributed in the whole sample, while Cu and Ni elements aggregate into granular forms, corresponding to the Cu–Ni alloy (Fig. 1g). The Cu and Ni contents were determined to be 37.8 and 33.5 wt%, respectively, as determined by inductively coupled plasma-mass spectrometry (ICP-MS), respectively. The contents of C and N are 23.2 and 4.2 wt%, respectively, as determined by elemental analysis. The product was denoted as Cu\(_{1.0}\)Ni\(_{1.0}\)/N–C. The chemical compositions and elemental states of the Cu\(_{1.0}\)Ni\(_{1.0}\)/N–C catalyst were investigated by X-ray photoelectron spectroscopy (XPS). In the high-resolution Cu 2p spectrum (Fig. 2a), the binding energies at 934.8 eV and 954.9 eV correspond to Cu\(^0\), while those at 932.8 eV and 952.6 eV can be assigned to Cu\(^2+\) and possibly existing Cu\(^+\).\(^{27,28}\) The Auger Cu LMM spectrum shows a peak at a binding energy of 918.5 eV (Fig. S4†), which excludes the presence of Cu\(^+\) species.\(^{28}\) In the high-resolution Ni 2p spectrum (Fig. 2b), the binding energies at 852.7 eV, 870.1 eV and 855.9 eV, 873.5 eV correspond to Ni\(^0\) and Ni\(^2+\), respectively.\(^{29,30}\) The four nitrogen species peaks in the high-resolution N 1s spectrum (Fig. 2c) correspond to pyridinic N (B.E. 398.9 eV), Cu/Ni–N (B.E. 399.5 eV), pyrrolic N (B.E. 400.9 eV) and graphitic N (B.E. 402.1 eV), respectively.\(^{9,31}\) This indicates that the N species on the catalyst surface is mainly in the pyridinic and pyrrolic form, and there is a small amount of the Cu/Ni–N form. The presence of graphitic N species indicates that the 3D network is mainly composed of nitrogen-doped graphene.\(^{32}\) Furthermore, synchrotron extended X-ray absorption fine structure (XAFS) analysis was performed to further investigate the alloy structure of Cu\(_{1.0}\)Ni\(_{1.0}\)/N–C. The Cu–Cu bond distance of Cu\(_{1.0}\)Ni\(_{1.0}\)/N–C shifts to 2.209 Å, which is lower than that of Cu foil (2.240 Å). However, the Ni–Ni bond distance of Cu\(_{1.0}\)Ni\(_{1.0}\)/N–C is 2.175 Å, which is a higher bond distance than that of Ni foil (2.082 Å). This phenomenon also proves the existence of the Cu/Ni alloy in Cu\(_{1.0}\)Ni\(_{1.0}\)/N–C (Fig. S5†).

A series of Cu\(_x\)Ni\(_y\)/N–C catalysts with different Cu to Ni molar ratios were synthesized and characterized (Fig. S6–S9 and Table S1†), where \(x/y\) represents the molar ratio of Cu to Ni. All the catalysts present similar morphologies to Cu\(_{1.0}\)Ni\(_{1.0}\)/N–C, i.e., the Cu/Ni alloy nanoparticles are embedded in the nitrogen-carbon network. The X-ray diffraction peaks of the Cu\(_x\)Ni\(_y\)/N–C catalysts shift toward a lower diffraction degree with increasing Cu content, implying the merging of more Cu atoms into the Ni lattice (Fig. S3†).\(^{15,32}\) For comparison, Cu/N–C and Ni/N–C catalysts were synthesized using a method similar to that for Cu\(_x\)Ni\(_y\)/N–C synthesis in the absence of nickel(ii) nitrate hexahydrate and copper powder, respectively (Fig. S10 and 11†).

The electrocatalytic activities of Cu\(_x\)Ni\(_y\)/N–C for the CO\(_2\) RR were studied in a two-compartment cell. To assess the CO\(_2\) electroreduction performance of Cu\(_x\)Ni\(_y\)/N–C, linear sweep voltammetry (LSV) measurements were performed in N\(_2\) and CO\(_2\) saturated 0.5 M KHCO\(_3\), respectively (Fig. 3a). For Cu\(_{1.0}\)Ni\(_{1.0}\)/N–C and Cu\(_{4.8}\)Ni\(_{1.0}\)/N–C, the increase of current density relates to the HER in the N\(_2\) saturated solution, and the combination of the CO\(_2\) RR and HER in the CO\(_2\) saturated solution.\(^{27}\) The sharp increase of current density in the CO\(_2\) saturated solution compared to that in the N\(_2\) saturated solution indicates that Cu\(_{1.0}\)Ni\(_{1.0}\)/N–C and Cu\(_{4.8}\)Ni\(_{1.0}\)/N–C are more favorable for the CO\(_2\) RR than the HER. However, the current density of Cu/N–C in CO\(_2\) saturated 0.5 M KHCO\(_3\) is close to that in the N\(_2\) saturated solution. This implies that the performance of Cu/N–C for
CO₂ reduction is lower than that of Cu₁₀Ni₁₀/N-C and Cu₄.₈Ni₁₀/N-C catalysts. Moreover, the current density of Cu₁₀Ni₁₀/N-C (40.3 mA cm⁻²) and Cu₄.₈Ni₁₀/N-C (30.1 mA cm⁻²) at −1.0 V is much higher than that of Cu/N-C (15.5 mA cm⁻²).

The gaseous products and liquid products formed in CO₂ reduction were analyzed by gas chromatography (GC) and ¹H nuclear magnetic resonance (¹H NMR) spectroscopy, respectively. The faradaic efficiencies of CO formation during the CO₂ RR at different applied potentials are presented in Fig. 3b. CO and H₂ were produced when catalyzed by Cu/N-C, and there is no liquid product in the whole electrolysis process. In contrast, H₂ is the main catalytic product with a small amount of CO and HCOOH when catalyzed by Cu/N-C, and H₂ is the only catalytic product when catalyzed by Ni/N-C (Fig. S12–S14†).

Outstandingly, the maximum faradaic efficiency FE(CO) of Cu₁₀Ni₁₀/N-C is 94.5% at a low potential of −0.60 V, with a high current density of 18.8 mA cm⁻². In sharp contrast, the FE(CO) of Cu/N-C is only 20.4% at the same potential, with a small current density of 6.5 mA cm⁻². Such a high FE(CO) of Cu₁₀Ni₁₀/N-C at low potential provides a maximum energy conversion efficiency of 69.2%, which is about 4.6 times that of Cu/N-C (Fig. S15†).³⁸,³⁹ Compared with the reported Cu-based alloys and bimetallic catalysts, the catalytic performance of Cu₁₀Ni₁₀/N-C is superior owing to the combined high FE(CO) and current density at relatively low potential (Table S2†).³⁸,³⁹,⁴⁰,⁴¹

Fig. 3c shows the total current density and partial current density of different catalysts at −0.60 V for CO formation. Cu₁₀Ni₁₀/N-C has a J₉₀ of 17.7 mA cm⁻² at −0.60 V, which is about 13.4 times that of the Cu/N-C catalyst (1.32 mA cm⁻²). Moreover, the partial current density for CO formation increases first and then decreases with applied potential (Fig. S16†), indicating that high applied potentials can accelerate the reaction rate for CO₂ reduction. Attractively, the catalytic performance of Cu₁₀Ni₁₀/N-C is very stable, because the current density and FE(CO) remain undiminished after 38 h of constant potential electrolysis (Fig. 3d). The reused Cu₁₀Ni₁₀/N-C catalyst was characterized by different techniques (Fig. S17 and 18†). It is observed that the Cu₁₀Ni₁₀/N-C catalyst is very stable against long-term electrolysis.

The electrochemical surface area (ECSA), which can be evaluated by comparing the double-layer capacitance (Cdl) of catalysts, is an important factor affecting the catalytic performance. From Fig. 4a, the Cdl of Cu₁₀Ni₁₀/N-C is about 2 times that of Cu/N-C. The large ECSA will provide abundant active sites, and thus can enhance the activity for the CO₂ RR. The Tafel slopes of Cu₁₀Ni₁₀/N-C and Cu₄.₈Ni₁₀/N-C are 113.4 and 104.2 mV dec⁻¹, respectively (Fig. 4b), which are close to the theoretical value of 116 mV dec⁻¹. It indicates that the single electron transfer to CO₂ to form the surface adsorbed CO₂⁻ intermediate is the rate-determining step for Cu₂⁻Ni₂⁻/N-C. The values of Cu₄Ni₁₀/N-C are obviously lower than those of Cu/N-C, implying that the formation of CO by Cu₄Ni₁₀/N-C is more favorable kinetically. Electrochemical impedance spectroscopy (EIS) was used to further explain the reaction kinetics of the CO₂ RR. EIS was carried out in 0.5 M KHCO₃ solution at an open circuit potential (Fig. 4c), and the equivalent circuit (Fig. S19†) was then used to fit the impedance data. The Nyquist plots confirm that Cu₁₀Ni₁₀/N-C and Cu₄.₈Ni₁₀/N-C possess smaller charge transfer resistance than Cu/N-C. This indicates a faster electron transfer to CO₂ for forming the reduced CO₂⁻ intermediate in the CO₂ RR catalyzed by Cu₄Ni₁₀/N-C, thus leading to greatly enhanced catalytic efficiency for CO₂ reduction.⁴⁵

The above results reveal that the Cu₄Ni₁₀/N-C catalyst has superior electrocatalytic performance for CO₂ reduction to CO, in particular compared with mono-metal catalysts. In
In a typical synthesis process, appropriate amounts of copper and nickel nitrate hexahydrate were dispersed in 10 mL of anhydrous methanol and stirred at room temperature for 10 min. After this, the solution was transferred into a Teflon-lined stainless-steel autoclave, and the autoclave was sealed and heated at 120 °C for 24 h under stirring. After the autoclave cooled to room temperature, the as-prepared sample was washed with methanol and dried in a vacuum oven at 80 °C for 24 h. The solid was annealed in a tube furnace at 900 °C in a N₂ atmosphere for 1 h with a heating rate of 2 °C min⁻¹.

**Characterization**

The morphologies of the catalysts were characterized by SEM (HITACHI S-4800). TEM images were taken on JEOL JEM-1011 and JEOL JEM-2100F field-emission transmission electron microscopes operated at 200 kV. XRD patterns were recorded on a Rigaku D/max 2400 diffractometer with Cu Kα radiation (λ = 0.15418 nm) with a scanning rate of 4° min⁻¹. XPS was carried out with a multipurpose X-ray photoelectron spectrometer (Thermo Scientific ESCALAB 250Xi). The elemental analysis of C, N, and O was performed on a FLASH EA1112 elemental analysis instrument. The contents of Cu and Ni elements were determined by ICP-MS. The XAFS experiment was carried out at Beamline 1W1B at BSRF. Data of XAFS were processed using the Athena and Artemis programs of the IFEFFIT package based on FEFF 6. Data were processed with k²-weighting and an Rbkg value of 1.0. Normalized XANES data were obtained directly from the Athena program of the IFEFFIT package.

**Fabrication of electrodes**

The working electrodes were prepared by loading the sample suspension onto carbon paper (CP). Briefly, the as-prepared catalysts and 10 μL Nafion D-521 dispersion were dispersed in absolute ethanol and ultrasonicated for 30 min to form a uniform suspension, and the suspension was loaded on 1 cm x 1 cm CP. The electrode was dried in a vacuum oven at 80 °C for 6 h before electrochemical experiments.

A CHI660E electrochemical analyzer (Shanghai Chenhua Instrument Co. LTD, China) was used in all the electrochemical experiments. A thre-electrode test was carried out in a sealed H-cell which was separated by a Nafion N117 membrane. The reference and counter electrodes are Ag/AgCl and a Pt net (1 x 1 cm), respectively. All experiments were carried out at atmospheric pressure and room temperature. Linear sweep voltammetry (LSV), amperometric i-t curve measurements and electrochemical impedance spectroscopy (EIS) were performed in 0.5 M KHCO₃ solution. Before experiments, the electrolyte solution in the working compartment was bubbled for 1 h with N₂ (99.99%) and CO₂ (99.999%) to form a saturated solution. The pH values of the electrolyte solutions with N₂ and CO₂ are 8.8 and 7.3, respectively. All potentials reported in this paper are with respect to the reversible hydrogen electrode (RHE), which were converted using eqn (1).

Potential in the RHE = applied potential vs. Ag/AgCl

\[ +0.21 \text{ V} + 0.0592 \times \text{pH} \]  

(1)

The gaseous product in the gas bag was collected and analyzed using a gas chromatograph (GC, HP 4890D), which was equipped with TCD and FID detectors, using helium as the internal standard. The liquid mixture was analyzed by the ¹H-NMR method, and the spectra were recorded on a Bruker
Avance III 400 HD spectrometer in deuterium oxide-d2 with TMS as an internal standard.

**Electrochemical impedance spectroscopy (EIS)**
The EIS measurement was carried out in 0.5 M KHCO3 solution at an open circuit potential (OCP) with an amplitude of 5 mV in the frequency range of 10^-2 to 10^7 Hz.

**Double-layer capacitance (Cdl)**
The Cdl was determined by measuring the capacitive current associated with double-layer charging from the scan-rate dependence of cyclic voltammetric stripping. The scan rates were 5 mV s^-1, 10 mV s^-1, 15 mV s^-1, 20 mV s^-1, 25 mV s^-1, 30 mV s^-1, 40 mV s^-1 and 50 mV s^-1.

**Energy efficiency**
The energy efficiency of reduction of CO2 to CO was obtained using eqn (2):

$$\eta_{\text{CO}} = \frac{\text{FE(CO)} \times \Delta E_{\text{CO}}}{\Delta E_{\text{CO}}}$$

(2)

In the equation, $\eta_{\text{CO}}$ stands for the cathodic energy conversion efficiency of the CO formation; $\Delta E_{\text{CO}}$ represents the difference between the standard half reaction potentials for water oxidation (1.23 V vs. RHE) and reduction of CO2 to CO (−0.11 V vs. RHE); $\Delta E_{\text{CO}}$ stands for the difference between the standard water oxidation potential and the applied potential for CO2 reduction.

**Conflicts of interest**
The authors of this manuscript have no conflicts of interest.

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