Electronic Supplementary Information

Achieving 59% Faradaic Efficiency of N$_2$ Electroreduction Reaction in Aqueous Zn-N$_2$ Battery by Facilely Regulating the Surface Mass Transport on Metallic Copper

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Experimental

**Chemicals:** Carbon paper (CP) was purchased from TORAY Group Co. Ltd. $^{15}$NH$_4$Cl, NaClO aqueous solution, sodium nitroprusside (C$_3$FeN$_6$Na$_2$O) and p-Dimethylaminobenzaldehyde were purchased from Aladdin Chemical Co., Ltd. Other chemicals (H$_2$SO$_4$, HCl, CuSO$_4$, NaOH, KOH, NH$_4$Cl, N$_2$H$_4$·H$_2$O, acetone, sodium citrate dehydrate, salicylic acid and ethanol were received from Beijing Chemical Works. Dimethyl sulfoxide-d (99.8 atom% D) with 0.03% (V/V) TMS was purchased from INNO-Chem. Water was obtained by a water purification system (18.25 MΩ cm). All reagents were used without further purification.

**Materials preparation:** Before the electrodeposition process, CP was fully washed by acetone and diluted HCl solution. The electrodeposition of Cu nanoparticles was conducted using three-electrode system with a piece of CP served as working electrode. A saturated calomel electrode (SCE) and a graphite rod were used as reference and counter electrodes, respectively. The electrodeposition was carried out using galvanostatic technique under the current of 10 mA (current density of 10 mA cm$^{-2}$) in 0.5 M H$_2$SO$_4$ + 0.05 M CuSO$_4$ with the CP as substrate. Under the galvanostatic condition, Cu$^{2+}$ in the solution can be reduced on the CP substrate to gradually form Cu nanoparticles. The as-synthesized samples are denoted as Cu-t, t represents the electrodeposition time, i.e. 2, 10, 30 and 60 min for Cu-2, Cu-10, Cu-30, and Cu-60, respectively. To avoid the oxidation of Cu nanoparticles, the obtained samples were stored in Ar atmosphere or directly used for NRR test.

**Material characterization:** Scanning electron microscopy (SEM) images were captured on a Hitachi S4800 with a working accelerating voltage of 10 kV. X-ray diffraction (XRD) was measured on a Rigaku Dmax 2500 equipped with Cu-Kα radiation. Inductively coupled plasma-atomic emission spectrometry (ICP-AES) tests were carried on X Series 2, Thermo Scientific USA). UV-Vis absorption spectroscopy was tested on MAPADA UV-3000PC Spectrophotometer.

**Electrochemical NRR measurements:** All electrochemical tests were measured on CHI 660B electrochemical
workstations at room temperature with 95% iR corrected. A three electrode system were fabricated with the prepared Cu-based materials, platinum wire and saturated calomel electrode (SCE) severing as working electrode, counter electrode and reference electrode, respectively. Platinum wire was in the anode compartment alone to avoid the electrochemical oxidation of produced NH$_3$. Before NRR tests, the 211 Nafion membrane was firstly activated in 5% H$_2$O$_2$, H$_2$O, 0.5 M H$_2$SO$_4$ and H$_2$O for 1 h at 80 °C, respectively. The synthesized Cu-based catalyst directly served as working electrode with a Pt electrode holder, Pt wire and saturated calomel electrode (SCE) worked as counter electrode and reference electrode, respectively. Notably, the deposited Cu nanoparticles are very easily oxidized in air, thus the Cu foil after electrodeposition was quickly transferred into N$_2$- or Ar-saturated electrolyte for NRR tests. A gas-tight H-type electrochemical cell equipped with a piece of 211 Nafion membrane was employed to conduct the electrochemical reaction, as shown in Fig. S3. The NRR catalytic activities were evaluated using potentiostatic technique under selective potential for 2 h.

**Electrochemical surface area (ECSA) measurement**: The ECSA of the Cu-based samples was measured by the double-layer capacitance method. Typically, the cyclic voltammetry (CV) test was conducted under the potential range of 0.75-0.80 V (vs. RHE) with scan rates of 20, 40, 60, 80 and 100 mV s$^{-1}$. The charging currents were plotted against the scan rates, and the slope of the obtained linear regression represents the capacitance, as presented in Fig. S6. For better comparison of the activities of the prepared Cu-based catalysts, the ECSAs of all the samples were normalized to the ECSA of Cu-2.

**Determination of ammonia**: The concentration of NH$_3$ produced from electrocatalysis was determined by the indophenol blue method (Nature Mater. 2013, 12, 836) with slight modification. In detail, 2 mL of the electrolyte was taken out after electrocatalytic reaction, and added into 2 mL of a 1 M NaOH solution containing salicylic acid and sodium citrate. Then, 1 mL of 0.05 M NaClO and 0.2 mL of 1 wt% C$_3$FeN$_6$Na$_2$O were also added into the above mixed solution. The UV-Vis absorption spectrum was measured after 2 hours. The concentration of
indophenol blue was determined using the absorbance at the wavelength of 655 nm. The concentration-absorbance curves were calibrated using standard ammonia chloride solution with a series of concentrations in 0.1 M KOH. The fitting curve \((y = 0.477x + 0.001, R^2=0.999)\) shows good linear relationship between the absorbance values and \(\text{NH}_3\) concentrations by three times independent calibrations.

**Determination of hydrazine:** The hydrazine product in the electrolyte was analyzed by the method of Watt and Chrisp (Anal. Chem. 1952, 24, 2006). Briefly, 5.99 g of p-dimethylaminobenzaldehyde, 30 mL of concentrated HCl and 300 mL ethanol were mixed as a color reagent. To determine the hydrazine in the electrolyte, 5 mL electrolyte was taken out and mixed with 5 mL of above color reagent. After 10 min, the absorbance of the solution at 455 nm was measured, and the yield of hydrazine was estimated from a concentration-absorbance standard curve. The standard curve was calibrated using standard hydrazine monohydrate solutions with a series of concentrations in 0.1 M KOH. The fitting curve \((y = 0.781x + 0.003, R^2=0.999)\) shows a good linear relation of absorbance value with hydrazine concentration by three times independent calibrations.

**Calculations of the \(\text{NH}_3\) formation rate and Faradaic efficiency (FE):** The Faradaic efficiency for \(\text{NH}_3\), geometric and normalized \(\text{NH}_3\) synthesis rates \((R)\) are calculated as follows:

\[
\text{Geometric } R_{\text{NH}_3} = \frac{c_{\text{NH}_3} \times V \times M_{\text{NH}_3}}{A \times t}
\]

\[
\text{Normalized } R_{\text{NH}_3} = \frac{c_{\text{NH}_3} \times V \times M_{\text{NH}_3}}{A \times t \times \frac{\text{ECSA}}{\text{ECSA}_{\text{Cu}-0}}}
\]

\[
\text{Faradaic Efficiency} = \frac{c_{\text{NH}_3} \times V \times 3 \times F}{\int_0^t idt}
\]

Where \(c_{\text{NH}_3}\) is the determined \(\text{NH}_3\) concentration;

\(V\) is the volume of electrolyte in the cathode compartment, typically 60 mL;

\(M_{\text{NH}_3}\) is the molecular weight of ammonia, 17 g mol\(^{-1}\);

\(A\) is the geometric surface area of the electrode, 1 cm\(^2\);
ECSA could be represented by the slope of the linear regression;

$t$ is the time of electrolysis, 7200 s;

$F$ is the Faraday constant, i.e., $96485 \text{ C mol}^{-1}$.

The reported values of $R$ and FE were calculated based on three separately measurements under the same conditions.

**Electrochemical in situ FTIR experiments:** Before measurement, the N$_2$/Ar was passed into electrolyte for 30 minutes and continuously fed into the electrolyte during test. A Nicolet iS50 FTIR spectrometer equipped with a liquid-nitrogen-cooled MCT-B detector was used to measure the in situ FTIR spectroscopy. A CaF$_2$ disk is used as the IR window. Every spectrum is collected by 512 times scan under the spectral resolution of 4 cm$^{-1}$. The spectra were presented as the relative change in reflectivity based on $\Delta A = -\log[R(E1)/R(E2)]$, $\Delta A$ is relative absorbance, $R(E1)$ and $R(E2)$ are the electrode reflectivity at potentials $E1$ and $E2$ (reference), respectively.

**$^{15}$N$_2$ isotope labeling experiments:** The isotopic labeling experiment was conducted using $^{15}$N$_2$ as the feeding gas in 0.1 M KOH. After electrolysis at 0 V for 2 h, the electrolyte was taken out and acidized to pH ~3 by adding 1 M HCl. After lyophilization, the obtained solid ammonium salt was dissolved in 0.5 mL dimethyl sulfoxide-d for $^1$H nuclear magnetic resonance measurement ($^1$H NMR, Bruker Avance III 600 MHz).

**Calculation details:** Density functional theory (DFT) calculations were performed based on Vienna ab-initio Simulation Package (VASP).$^1$ The geometry and electronic structures were optimized with the generalized gradient approximation (GGA) of Perdew–Burke–Ernzerhof (PBE) to represent the exchange-correlation energy.$^1$ Core electrons effect on the valence electron density was treated by using Projector Augmented Wave (PAW) method.$^2$ The cutoff energy for the plane wave basis sets was 400 eV. The convergence threshold of atoms position and cell parameters were set to be $10^{-5}$ eV in energy and $10^{-2}$ eV/Å in force. The Brillouin zone was sampled by k-points mesh of $4^*4^*1$ for the Cu (220) surface slab.$^3$ The van der Waals (vdW) interaction was calculated by DFT-D3
Combined with the XRD results, we studied the NRR processes on the three crystal faces of Cu (111) (200) and (220). The calculated lattice parameter of the Cu bulk structure is $a = 3.64 \text{ Å}$. The optimized lattice of bulk Cu was used to construct slabs for Cu (111), Cu (200) and Cu (220) surfaces. A four-layer slab containing 3*3 unit cells represents the Cu (111), Cu (200) and Cu (220) surfaces. The vacuum space with a thickness more than 15 Å was set to avoid the interactions of the periodic images. In calculations, the bottom two layers of the slab were fixed, and all other atoms were fully relaxed.

The Gibbs free energy calculation was based on the method of Nørskov et al. The chemical potential of a proton and electron pair ($\mu (\text{H}^+ + e^-)$) is equal to a half of gaseous hydrogen ($\mu (\text{H}_2)$). For each elemental step, the Gibbs free energy ($\Delta G$) was calculated based on the following equation:

$$\Delta G = \Delta E_{\text{DFT}} + \Delta ZPE + \Delta H - T \Delta S + \Delta G_{\text{PH}} + \Delta G_U,$$

where $E$ is the total energy, ZPE is the zero point energy, $H$, $S$ are the heat capacity and entropy, $T$ is equal to 298 K. $G_{\text{PH}}$ and $G_U$ are the influence caused by pH value and electrode potential $U$, respectively.

$$G_U = -neU,$$

where $n$ is the transferred electron number.

$$G_{\text{PH}} = -k_B T \ln[H^+] = \text{pH} \times k_B T \ln 10,$$

where $k_B$ is the Boltzmann constant.

The adsorption energies ($E_{\text{ad}}$) of the different adsorbates were calculated by the expression:

$$E_{\text{ad}} = E_{\text{total}} - (E_{\text{catal}} + E_{\text{mol}})$$

where $E_{\text{total}}$, $E_{\text{catal}}$, $E_{\text{mol}}$ are the total energy of slab after adsorption, bare catalyst, and the adsorbate molecule including the $N_2$, $N_xH_y$, respectively.
Fig. S1 SEM image of the carbon paper (CP) before electrodeposition treatment. The diameter of the carbon fibres is about 8 μm; the inset shows the photograph of the CP on an electrode holder.
Fig. S2 SEM images of (A) Cu-0.5; (B) Cu-1; (C) Cu-2.

As seen in Fig. S2, if the electrodeposition time is less than 2 min, most of the carbon fiber surface can not be covered by Cu nanoparticles, which leads to low utilization of carbon paper and low catalytic activity.
Fig. S3 XRD spectrum of the as-prepared Cu-2 catalyst. Three characteristic diffraction peaks at 43.3°, 50.4° and 74.1° are apparently observed and can be assigned to (111), (200) and (220) crystal faces of Cu, respectively. The sharp peaks at 26° and 54° are ascribed to the graphite-2H phase of the CP substrate.
Fig. S4 The photograph of a H-type electrochemical cell used for NRR tests.
Fig. S5 UV-Vis spectra and calibration curves for NH$_3$ (A and B) and N$_2$H$_4$ (C and D) in 0.1 M KOH.
Fig. S6 The i-t curves from the blank CP (A) and the corresponding UV-Vis spectra (B-C) of electrolyte after electrolyzation in N$_2$-saturated and Ar-saturated 0.1 M KOH at varied potentials.
Fig. S7 (A) UV-Vis spectra of electrolyte after electrolyzation in 0.1 M KOH saturated with N₂ at different potentials and saturated with Ar at 0.0 V on Cu-2; (B) UV-Vis spectra of electrolyte after electrolyzation in 0.1 M KOH saturated with Ar at different potentials on Cu-2.
Fig. S8 (A) LSV curves of Cu-2 in N$_2$- and Ar-saturated 0.1 M KOH, scan rate: 2 mV s$^{-1}$; (B) The enlarged LSV curves of the marked part in (A).

In this study, the potential window was chosen according to the LSV test, as shown in Fig. S7. Clearly, in the LSV curves of Cu-2 under N$_2$ and Ar conditions, the N$_2$ reduction current signal first appears at about 0 V. At more positive potentials, the currents in Ar and N$_2$ are almost same, indicating no N$_2$ reduction. However, the current in N$_2$ condition is much larger than that in Ar under the more negative potentials. Hence, we chose the potential window below 0 V to study the NRR catalytic performance of the samples.
**Fig. S9** i-t curves from the Cu-based catalysts and the corresponding UV-Vis spectra of electrolyte after electrolyzation in 0.1 M KOH saturated with N₂ at varied potentials and saturated with Ar at 0 V.
Fig. S10 CV curves and the calculated ECSAs of the Cu-based catalysts, in Ar-saturated 0.1 M KOH under the potential range of 0.75-0.80 V with potential scan rates of 20, 40, 60, 80 and 100 mV s\(^{-1}\).
Fig. S11 The geometric R (A) and ECSA normalized R (B) of the Cu-based catalysts.
Fig. S12 (A) EIS spectra of the Cu-based catalysts under 0.0 V in Ar-saturated 0.1 M KOH; (B) ECSA-normized HER activities of the Cu-based catalysts in Ar-saturated 0.1 M KOH with a potential scan rate of 5 mV s$^{-1}$. 
Such an integrated cell consists of NRR-catalyst cathode, Zn metal anode and aqueous alkaline electrolyte (0.1 M KOH). It can be seen that such Zn-N₂ cell is very simple but it can utilize the chemical energy of Zn metal to synthesize NH₃ from the N₂ and H₂O, as well as generate electricity. To examine the ability of such Zn-N₂ cell for NH₃ synthesis, the cell was discharged at 1 μA cm⁻² for 1 h. An average NH₃ synthesis rate of 0.125 μg h⁻¹ cm⁻² was determined based on four repeated tests. The higher FE in Zn-N₂ cell than that of obtained in H-type cell should be ascribed to the smaller over potential with less HER competition on the surface of Cu. Also, the low NH₃ synthesis rate is reasonable due to the small current through the catalyst surface.
Fig. S14 The optimized geometry structure of N₂ adsorption on Cu (111) and (200).
Fig. S15 Partial density of states (PDOS) of N₂ adsorbed on Cu (220). There is an effective overlap between the N-2p orbitals and Cu-3d orbitals around 2 eV and -2 eV below the Fermi level, indicating the strong interaction of N₂ and Cu. The electrons of Cu were transferred into the anti-bond level of N₂, leading to the decrease of bond order in N₂, which agrees well with the previous structural calculations results.
Fig. S16 The *in situ* FTIR spectra of Cu-2 catalyst in Ar-saturated 0.1 M KOH. In the FTIR spectra under N\(_2\) condition, two positive broad peaks at 3628-3600 cm\(^{-1}\) and 1611-1607 cm\(^{-1}\) are identified as the H-O-H bending and O-H stretching of H\(_2\)O molecules, respectively (Chem. Commun. 2019, 55, 687). With the potential decreasing, the intensities of these two peaks increase due to the orientation change of H\(_2\)O molecules on the Cu surfaces (J. Am. Chem. Soc. 2018, 140, 1496). An obvious negative broad peak at 3409-3386 cm\(^{-1}\) is caused by the stretching of O-H group in the ice-like second water on Cu surface (J. Am. Chem. Soc. 2018, 140, 1496). These water-related peaks are also observed in the spectra collected in Ar condition, while the N-related peaks are only found in the N\(_2\) condition.
Fig. S17 The Gibbs free energy diagram of three possible paths for N\textsubscript{2} reduction reaction on Cu (220) at U = 0 V. Calculation results show that there are three possible reaction paths for the NRR over Cu surface. However, the path B (associate mechanism) is the most possible one due to its lowest energy barrier for N\textsubscript{2} activation.
Table S1. The ICP-AES results.

<table>
<thead>
<tr>
<th>Mass density (mg cm$^{-2}$)</th>
<th>Cu-2</th>
<th>Cu-10</th>
<th>Cu-30</th>
<th>Cu-60</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.09</td>
<td>0.45</td>
<td>1.3</td>
<td>2.5</td>
<td></td>
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</table>
Table S2. Summary of the catalytic performances of the recently reported catalysts toward NRR in aqueous under ambient condition.

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>electrolyte</th>
<th>R@E</th>
<th>FE @E</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-2</td>
<td>0.1 M KOH</td>
<td>1.4 µg h⁻¹·mg⁻¹</td>
<td>59% @ 1µA cm² in Zn-N₂ cell</td>
<td>This work</td>
</tr>
<tr>
<td>Cu-2</td>
<td>0.1 M KOH</td>
<td>12.0 µg h⁻¹·mg⁻¹</td>
<td>33.0% @0V</td>
<td>This work</td>
</tr>
<tr>
<td>Cu-10</td>
<td>0.1 M KOH</td>
<td>4.4 µg h⁻¹·mg⁻¹</td>
<td>11.0% @0V</td>
<td>This work</td>
</tr>
<tr>
<td>Cu-30</td>
<td>0.1 M KOH</td>
<td>1.1 µg h⁻¹·mg⁻¹</td>
<td>2.7% @0V</td>
<td>This work</td>
</tr>
<tr>
<td>Cu-60</td>
<td>0.1 M KOH</td>
<td>0.2 µg h⁻¹·mg⁻¹</td>
<td>1.47% @0V</td>
<td>This work</td>
</tr>
<tr>
<td>Defect-rich Bi</td>
<td>0.2 M Na₂SO₄</td>
<td>5.453 µg h⁻¹·mg⁻¹</td>
<td>11.68% at -0.6 V</td>
<td>Angew. Chem. Int. Ed. 10.1002/anie.201903969</td>
</tr>
<tr>
<td>MoS₂</td>
<td>0.1 M Na₂SO₄</td>
<td>29.28 µg h⁻¹·mg⁻¹</td>
<td>8.34% @−0.40 V</td>
<td>Adv. Energy Mater. 2018, 1801357</td>
</tr>
<tr>
<td>Au</td>
<td>0.1 M KOH</td>
<td>1.648 µg h⁻¹·cm²</td>
<td>~4 % @-0.2V</td>
<td>Adv. Mater. 2017, 29, 1604799</td>
</tr>
<tr>
<td>Mo₂C/C</td>
<td>HCl (pH=2 or 3)</td>
<td>11.3 µg h⁻¹·mg⁻¹</td>
<td>7.8% @-0.3V</td>
<td>Adv. Mater. 2018, 1803694</td>
</tr>
<tr>
<td>a-Au/Fe₂O₃–RGO</td>
<td>0.1M HCl</td>
<td>8.3 µg h⁻¹·mg⁻¹</td>
<td>10.10% @-0.2 V</td>
<td>Adv. Mater. 2017, 29, 1700001</td>
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<tr>
<td>carbon nitride</td>
<td>0.1M HCl</td>
<td>8.09 µg h⁻¹·mg⁻¹</td>
<td>11.59% @-0.2V</td>
<td>Angew. Chem. Int. Ed. 10.1002/anie.201805514</td>
</tr>
<tr>
<td>NCM–Au</td>
<td>0.1 M HCl</td>
<td>0.36 g m² h⁻¹·@-0.2V</td>
<td>22 % @-0.1V</td>
<td>Angew. Chem. Int. Ed. 10.1002/anie.201805514</td>
</tr>
<tr>
<td>palladium nanoparticles</td>
<td>0.1 M PBS</td>
<td>~4.5 µg h⁻¹·mg⁻¹</td>
<td>8.2% @0.1 V</td>
<td>Nat. Commun., 2018, 9, 1795</td>
</tr>
<tr>
<td>N,P,C</td>
<td>0.1 M HCl</td>
<td>0.97 µg h⁻¹·mg⁻¹</td>
<td>4.2% @-0.2V</td>
<td>Chem. Commun., 2019, 55, 687-690</td>
</tr>
<tr>
<td>B-C</td>
<td>0.05 M H₂SO₄</td>
<td>9.8 µg h⁻¹·mg⁻¹</td>
<td>10.8% @0.5 V</td>
<td>Joule, 2018, 2, 1610</td>
</tr>
<tr>
<td>N-doped carbon</td>
<td>0.1 M KOH</td>
<td>15.7 µg h⁻¹·mg⁻¹</td>
<td>1.45% @-0.2 V</td>
<td>J. Mater. Chem. A, 2018, 6, 7762</td>
</tr>
<tr>
<td>TiO₂·rGO</td>
<td>0.1 M Na₂SO₄</td>
<td>15.13 µg h⁻¹·mg⁻¹</td>
<td>3.3% @-0.90 V</td>
<td>J. Mater. Chem. A, 2018, 6, 17303</td>
</tr>
<tr>
<td>CoP</td>
<td>1.0 M KOH</td>
<td>10.78 µg h⁻¹·mg⁻¹</td>
<td>7.36% @0 V</td>
<td>Small Methods 2018, 1800204</td>
</tr>
<tr>
<td>NbO₂</td>
<td>0.05 M H₂SO₄</td>
<td>11.6 µg h⁻¹·mg⁻¹</td>
<td>32% @-0.60 V</td>
<td>Small Methods 2018, 1800386</td>
</tr>
<tr>
<td>PdCo/CuO</td>
<td>0.1M KOH</td>
<td>10.04 µg h⁻¹·mg⁻¹</td>
<td>2.16% @-0.2V</td>
<td>Nanoscale, 2018, DOI: 10.1039/C8NR08724E</td>
</tr>
<tr>
<td>Au nanocages</td>
<td>0.5 M LiClO₄</td>
<td>3.74 µg h⁻¹·cm⁻²</td>
<td>35.9% @-0.4 V</td>
<td>J. Phys. Chem. Lett. 2018, 9, 5160</td>
</tr>
<tr>
<td>Au SAs-NDPCs</td>
<td>0.1 M HCl</td>
<td>2.32 µg h⁻¹·cm⁻²</td>
<td>12.3% @-0.2 V</td>
<td>Small Methods 2018, 1800202</td>
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</table>
Table S3. The adsorption energy, N-N bond and transferred charge of \( \text{N}_2 \) adsorption on Cu (111), Cu (200) Cu (220).

<table>
<thead>
<tr>
<th>Crystal face</th>
<th>( E_{ad} )/eV</th>
<th>N-N bond/Å</th>
<th>Transferred charge / e</th>
</tr>
</thead>
<tbody>
<tr>
<td>111</td>
<td>-0.13</td>
<td>1.118</td>
<td>0.04</td>
</tr>
<tr>
<td>200</td>
<td>-0.14</td>
<td>1.118</td>
<td>0.04</td>
</tr>
<tr>
<td>220</td>
<td>-0.41</td>
<td>1.127</td>
<td>0.1</td>
</tr>
</tbody>
</table>

References


