# **Electronic Supplementary Information**

### **Experimental Section**

Materials: Zirconium oxychloride (ZrOCl·8H<sub>2</sub>O, 99.0%), poly-(vinyl pyrrolidone) (PVP, M<sub>W</sub> =1300000), dimethyl formamide (DMF), boron (B, 99.0%), sulphur (S, 99.0%), sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>, 99.0%), hydrochloric acid (HCl, 99.0%), ammonium chloride (NH<sub>4</sub>Cl), 99.0%), salicylic acid ethanol  $(C_2H_6O,$  $(C_7H_6O_3),$ sodium citrate dehydrate  $(C_6H_5Na_3O_7 \cdot 2H_2O)$ , p-dimethylaminobenzaldehyde  $(C_9H_{11}NO)$ , sodium nitroferricyanide dihydrate (C<sub>5</sub>FeN<sub>6</sub>Na<sub>2</sub>O·2H<sub>2</sub>O) and sodium hypochlorite solution (NaClO) were purchased from Aladdin Ltd. (Shanghai, China). Nafion (5 wt%) solution was purchased from Sigma-Aldrich Chemical Reagent Co., Ltd. Nitric acid (HNO<sub>3</sub>), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), hydrogen peroxide  $(H_2O_2)$ , hydrazine monohydrate  $(N_2H_4 \cdot H_2O)$  and ethyl alcohol  $(C_2H_5OH)$  were purchased from Beijing Chemical Corp. (China). chemical Ltd. in Chengdu. The ultrapure water used throughout all experiments was purified through a Millipore system. All reagents were analytical reagent grade without further purification.

**Preparation of ZrO<sub>2</sub> nanofiber:** 1.2 g PVP was dissolved in 10 mL DMF and stirred rigorously for 10 h. After that, 1.0 g ZrOCl·8H<sub>2</sub>O was added into above solution and stirred for 10 h. The resulting solution was transferred into a 5 mL syringe. A high voltage of 20 kV was applied with a flow rate of 15  $\mu$ L min<sup>-1</sup> for electrospinning. Then, the collected nanofibers were sintered at 600 °C for 2 h under air atmosphere.

**Preparation of ZrS<sub>2</sub> NF-Vs:** 0.2 g boron powder and 0.3 g sulfur powder were placed in the bottom of a quartz tube. 0.1g  $ZrO_2$  was placed in a shorter silica tube with smaller diameter and sealed bottom, and this tube was put into above tube (Fig. S1).<sup>1</sup> And then the outer tube was fused in vacuum and annealing at 550 °C for 8 h.

**Characterizations:** Power XRD data were acquired by a LabX XRD-6100 X-ray diffractometer with a Cu Kα radiation (40 kV, 30 mA) of wavelength 0.154 nm (SHIMADZU, Japan). SEM images were collected on a GeminiSEM 300 scanning electron microscope (ZEISS, Germany) at an accelerating voltage of 5 kV. TEM images were acquired on a HITACHI H-8100 electron microscopy (Hitachi, Tokyo, Japan) operated at 200 kV. XPS measurements were performed on an ESCALABMK II X-ray photoelectron spectrometer using Mg as the exciting source. The absorbance data of spectrophotometer was measured on UV-Vis spectrophotometer. The EPR spectrum at X-band frequency (~9.85 GHz) was obtained with a Magnettech A300 EPR spectrometer at room temperature.

**Electrochemical measurements:** In this paper, we use a H-type electrolytic cell separated by a Nafion membrane which was protonated by boiling in ultrapure water,  $H_2O_2$  (5%) aqueous solution and 0.5 M  $H_2SO_4$  at 80 °C for another 2 h, respectively. A three-electrode configuration is used for electrochemical experiments using catalyst coated carbon paper as the working electrode, Ag/AgCl (filled with saturated KCl solution) as the reference electrode, and graphite rod as the counter electrode. The electrochemical experiments were carried out with an electrochemical workstation (CHI 660E) in N<sub>2</sub>-saturated 0.1 M HCl solution. The potentials reported in this work were converted to reversible hydrogen electrode (RHE) scale via calibration with the following equation: E (RHE) = E (vs. Ag/AgCl) + 0.256 V and the presented current density was normalized to the geometric surface area.

**Preparation of ZrS<sub>2</sub> NF-Vs /CP:** Carbon paper (CP) was pretreated in 0.05M H<sub>2</sub>SO<sub>4</sub>, and then was sequentially cleaned in C<sub>2</sub>H<sub>5</sub>OH and deionized water several times by sonication to remove the surface impurities. To prepare the ZrS<sub>2</sub> NF-Vs/CP, 10 mg ZrS<sub>2</sub> NF-Vs and 40  $\mu$ L 5 wt% Nafion solution were dispersed in 960  $\mu$ L water/ethanol (V<sub>water</sub>/V<sub>ethanol</sub>=1/3) followed by 0.5 h sonication to form a homogeneous ink. 10  $\mu$ L ink was loaded onto a CP (1 × 1 cm<sup>2</sup>) and dried under ambient condition (ZrS<sub>2</sub> NF-Vs loading: 0.1 mg cm<sup>-2</sup>).

**Determination of NH<sub>3</sub>:** Concentration of produced NH<sub>3</sub> was spectrophotometrically determined by the indophenol blue method.<sup>2</sup> 2 mL sample was removed from the cathodic chamber, then added into 1 mL of oxidizing solution containing NaClO ( $\rho_{Cl} = 4 \sim 4.9$ ), then added 2 mL coloring solution containing 1 M NaOH solution, 5% C<sub>7</sub>H<sub>6</sub>O<sub>3</sub> and 5% C<sub>6</sub>H<sub>5</sub>Na<sub>3</sub>O<sub>7</sub>·2H<sub>2</sub>O, and 200 µL catalyst solution (0.1 g Na<sub>2</sub>[Fe(CN)<sub>5</sub>NO]·2H<sub>2</sub>O diluted to 10 ml with deionized water) in turn. Absorbance measurements were performed after one hour at  $\lambda$ = 655 nm. The concentration-absorbance curves were calibrated using standard NH<sub>3</sub> solution with a serious of concentrations. The fitting curve (y = 0.344x + 0.0474, R<sup>2</sup> = 0.999) shows good linear relation of absorbance value with NH<sub>3</sub> concentration by three times independent calibrations.

**Determination of N<sub>2</sub>H<sub>4</sub>:** N<sub>2</sub>H<sub>4</sub> was estimated by the method of Watt and Chrisp.<sup>3</sup> The color reagent was a mixed solution of 5.99 g C<sub>9</sub>H<sub>11</sub>NO, 30 mL HCl and 300 mL C<sub>2</sub>H<sub>5</sub>OH. In detail, 5 mL electrolyte was removed from the electrochemical reaction vessel, and added into 5 mL prepared color reagent and stirred 15 min at 25 °C. The absorbance of such solution at the absorbance of 455 nm was measured to quantify the hydrazine yields with a standard curve of hydrazine (y = 0.695x + 0.0510, R<sup>2</sup> = 0.999).

**Determination of FE and V\_{NH3}:** FE was calculated by equation:

 $FE = 3F \times c_{NH3} \times V / 17 \times Q$ 

NH<sub>3</sub> yield (V<sub>NH3</sub>) was calculated using the following equation:

$$V_{\rm NH3} = (c_{\rm NH3} \times V) / (t \times m_{\rm cat.})$$

The amount of NH<sub>3</sub> was calculated as follows:

$$m_{\rm NH3} = [\rm NH_3] \times V$$

Where F is the Faraday constant (96500 C mol<sup>-1</sup>),  $c_{NH3}$  is the measured NH<sub>3</sub> concentration, V is the volume of the HCl electrolyte for NH<sub>3</sub> collection (35 mL), Q is the total quantity of applied electricity; t is the reduction time (2h) and m<sub>cat.</sub> is the catalyst mass (0.1 mg).

**Calculation of ECSA:** Electrochemical capacitance measurements were used to determine the active surface area of ZrS<sub>2</sub> NF-Vs/CP. To measure the electrochemical capacitance, the potential was swept between -0.054 V to -0.154 V with scanning rates of 20, 40, 60 and 80 mV s<sup>-1</sup>, respectively. The specific capacitance can be converted into an ECSA using the specific capacitance value for a flat standard with 1 cm<sup>-2</sup> of real surface area. The specific capacitance for a flat surface is generally found to be in the range of 20–60 µF cm<sup>-2</sup>. In the following calculations of TOF we assume 60 µF cm<sup>-2</sup>.

#### **ECSA calculation:**

$$A_{ECSA}^{ZrS_2} = \frac{183 \,\mu F \,cm^{-2}}{60 \,\mu F \,cm^{-2} per \,cm_{ECSA}^{-2}} = 3.05 \,cm_{ECSA}^{-2}$$

#### **TOF calculation:**

$$TOF = \frac{NH_{3} \text{ yield } \times \text{ geometric area } \times 17 \times N_{A}}{surfacs \text{ sites } \times A_{ECSA}}$$

Since the exact nitrogen binding sites are not known, we conservatively estimate the number of active sites as the total number of surface sites (only Zr atoms as possible active sites).

# Surface sites per real surface area:

Surfaces sites = 
$$\left(\frac{Atoms \ per \ unit \ cell}{Volume/unit \ cell}\right)^{\frac{2}{3}} = 5.319 \times 10^{15} atoms \ cm_{real}^{-2}$$

# **TON calculation:** TON= TOF× t

**Computational method:** The spin-polarized DFT calculations were performed by using the Vienna ab initio simulation package with the projected augment wave pseudopotential,<sup>4,5</sup> and the Perdew, Burke, and Ernzerhof functional.<sup>6</sup> The DFT + D3 method has been adopted to describe the weak van der Waals interactions.<sup>7</sup> The plane-wave basis set has been used with the kinetic cutoff energy of 450 eV. The convergence criteria for the total energy and the Hellmann-Feynman force were  $10^{-5}$  eV and  $0.02 \text{ eV}\text{Å}^{-1}$ , respectively. The ZrS<sub>2</sub> (101) (2 × 5) supercell was studied with a vacuum layer of ~ 15 Å. For such supercell, 2×1×1 Monkhorst-Pack grids<sup>8</sup> were used for the structural optimization. The calculations of the Gibbs free-

energy change for each elemental step adopt the computational hydrogen electrode model,<sup>9</sup> which can be obtained by the following equation

$$\Delta G = \Delta E + \Delta E_{\rm ZPE} - T \Delta S$$

The relevant total energy (*E*), zero-point energy ( $E_{ZPE}$ ), and entropy (*S*) of the adsorbed intermediates were obtained from DFT calculations (Table S2), while the thermodynamic corrections for the free molecules were taken from the NIST databases.<sup>10</sup>



**Fig. S1.** Reaction container for the sulfidation of ZrO<sub>2</sub> nanofiber from in situ formation of boron sulfides.



Fig. S2. SEM image of ZrO<sub>2</sub> nanofiber.



**Fig. S3.** TEM image of ZrO<sub>2</sub> nanofiber.



Fig. S4. XPS spectra in (a) Zr 3d and (b) S 2p regions for  $ZrS_2$  NF.



**Fig. S5.** EPR spectrum of  $ZrS_2$  NF-Vs at room temperature.



**Fig. S6.** (a) UV-Vis absorption curves of indophenol assays kept with different concentrations of  $NH_4^+$  ions for 2 h at room temperature. (b) A calibration curve used to estimate the concentration of  $NH_4^+$  concentration.



Fig. S7. (a) UV-Vis curves of various  $N_2H_4$  concentrations after incubated for 15 min at room temperature. (b) Calibration curve used for calculation of  $N_2H_4$  concentrations.



**Fig. S8.** (a) Ion chromatogram of  $NH_4Cl$  with different concentrations in 0.1 M HCl and (b) corresponding standard curve. (c) Ion chromatogram for the electrolytes at a series of potentials after electrolysis for 2 h. (d)  $NH_3$  yield of  $ZrS_2$  NF-Vs/CP at corresponding potentials.



Fig. S9. UV-Vis absorption spectra of the electrolytes estimated by the method of Watt and Chrisp before and after 2 h electrolysis in  $N_2$  atmosphere at -0.35 V.



**Fig. S10.** (a) CV for  $ZrS_2$  NF-Vs in the non-faradaic capacitance current range at scan rates of 20, 40, 60 and 80 mV s<sup>-1</sup>. (b) Corresponding capacitive currents at -0.10 V as afunction of scan rate for  $ZrS_2$  NF-Vs.



Fig. S11. Plot of TOF vs. potential for  $ZrS_2$  NF-Vs.



**Fig. S12.** UV-Vis absorption spectra of the electrolytes colored with indophenol indicator after 2 h electrolysis under different conditions.



Fig. S13. SEM image for  $ZrS_2$  NF-Vs after stability test.



Fig. S14.

pattern of ZrS<sub>2</sub> NF-Vs/CP after stability test.

XRD



Fig. S15. XPS spectra in (a) Zr 3d and (b) S 2p regions for  $ZrS_2$  NF-Vs after 24 h electrocatalysis.



Fig.	<b>S16.</b> STEM and	corresponding EDX	elemental mapping images	of Zr and S of ZrS <sub>2</sub> NF-
Vs		after	stability	test.



**Fig. S17.** Side and top views of (a) pristine  $ZrS_2$  (101) and (b)  $ZrS_2$ -Vs surfaces. The  $ZrS_2$  (101) surface slab model contains nine atomic layers, in which the bottom four ones are fixed to mimic the bulk. In (b), the rough position of the S vacancy is enclosed by a dashed circle.



**Fig. S18.** Side views of the most stable configuration for the  $N_2$  adsorption on (a) pristine  $ZrS_2$  (101) surface and (b, c)  $ZrS_2$ -Vs surface. The adsorption energies (in eV) and the nearest distances (in Å) between the  $N_2$  molecule and the surface atoms as well as the N-N bond lengths (in Å) are given.



Fig. S19 (a) Free energy (in eV) diagrams and (b) local atomic structures of the reaction intermediates for the NRR on  $ZrS_2$ -Vs surace under zero potential along the consecutive pathway.

Catalyst	Electrolyte	NH <sub>3</sub> yield	FE (%)	Ref.
ZrS <sub>2</sub> NF-Vs	0.1M HCl	$30.72 \ \mu g \ h^{-1} \ mg^{-1}_{cat.}$	10.33	This work
ZrO <sub>2</sub>	0.1 M HCl	24.74 µg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	5.0	11
CoS <sub>2</sub> -N/S-C	0.05 M H <sub>2</sub> SO <sub>4</sub>	$25 \ \mu g \ h^{-1} \ m g^{-1}_{cat.}$	25.9	12
sulfur-doped graphene	0.1 M HCl	27.3 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	11.5	13
Co-dopd MoS <sub>2-x</sub>	0.01 M H <sub>2</sub> SO <sub>4</sub>	$0.6 \text{ mmol } h^{-1} \text{ mg}^{-1}_{\text{cat.}}$	10	14
Fe <sub>3</sub> S <sub>4</sub>	0.1 M HCl	75.4 µg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	6.45	15
Bi <sub>4</sub> V <sub>2</sub> O <sub>11</sub> /CeO <sub>2</sub>	0.1 M HCl	23.21 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	10.16	16
La <sub>2</sub> Ti <sub>2</sub> O <sub>7</sub>	0.1 M HCl	25.15 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	4.55	17
В	0.1 M HCl	31.37 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	4.84	18
B <sub>4</sub> C	0.1 M HCl	26.57 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	15.95	19
BP	0.1 M HCl	26.42 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	12.7	20
Bi	0.1 M HCl	25.86 μg h <sup>-1</sup> mg <sup>-1</sup> cat.	10.08	21
Cu	0.1 M HCl	25.63 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	15.12	22
Co <sub>3</sub> O <sub>4</sub> @NC	0.1 M HCl	42.58 $\mu$ g h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	8.5	23
PTCA-rGO	0.1 M HCl	24.7 µg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	6.9	24
oxygen-doped graphene	0.1 M HCl	21.3 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	12.6	25
NiWO <sub>4</sub>	0.1 M HCl	40.05 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	19.32	26
B-doped graphene	0.05 M H <sub>2</sub> SO <sub>4</sub>	9.8 $\mu g h^{-1} m g^{-1}_{cat.}$	10.8	27
N-C@NiO/GP	0.1 M HCl	8.09 $\mu$ g h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	11.59	28
VN	0.05 M H <sub>2</sub> SO <sub>4</sub>	$3.3 \times 10^{-10} \text{ mol s}^{-1} \text{ cm}^{-2}$	2.25	29
PCN	0.1 M HCl	$3.01 \times 10^{-10} \text{ mo1 s}^{-1} \text{ cm}^{-2}$	1.15	30

Table S1. Comparison of electrocatalytic NRR performance for  $ZrS_2$  NF-Vs with other electrocatalysts tested in acidic electrolytes under ambient conditions.

Species	$E_{\rm ZPE}({\rm eV})$	TS (eV)
N <sub>2</sub>	0.15	0.58
*N-N	0.20	0.08
*N-*NH	0.48	0.09
*N-*NH2	0.84	0.08
*N	0.09	0.03
*NH	0.32	0.09
*NH <sub>2</sub>	0.67	0.06
*NH <sub>3</sub>	1.01	0.11
*NH-*NH	0.80	0.10
*NH-*NH <sub>2</sub>	1.14	0.14
*NH <sub>2</sub> -*NH <sub>2</sub>	1.50	0.13
$1/2H_2$	0.14	0.21
NH <sub>3</sub>	0.89	0.60

**Table S2.** The zero-point energy ( $E_{ZPE}$  in eV) and the product (*TS* in eV) of temperature (*T* = 298.15 K) and entropy (*S*) of the different species along the reaction pathway on ZrS<sub>2</sub>-Vs, where \* represents the adsorption site.

Potential (V)	Yield rate (GSA) (mol s <sup>-1</sup> cm <sup>-2</sup> )	Yield rate (ECSA) (mol s <sup>-1</sup> cm <sup>-2</sup> )
-0.30V	4.24017 × 10 <sup>-11</sup>	1.39022 × 10 <sup>-11</sup>
-0.35V	5.01905 × 10 <sup>-11</sup>	1.64559 × 10 <sup>-11</sup>
-0.40V	$2.04236 \times 10^{-11}$	6.69628 × 10 <sup>-12</sup>
-0.45V	$1.66332 \times 10^{-11}$	$5.45350 \times 10^{-12}$
-0.50V	9.42630 × 10 <sup>-12</sup>	$3.09059  imes 10^{-12}$

Table S3. ECSA and mass normalized  $\rm NH_3$  yield rates.

Atomic%	Zr	S
samples		
ZrS <sub>2</sub> NF-Vs	34.04	65.96
ZrS <sub>2</sub> NF-Vs after electrolysis	33.89	66.11

**Table S4.** Element analysis of  $ZrS_2$  NF-Vs by XPS data.

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