

## Electronic Supplementary Information

### Experimental Section

**Materials:** Bulk  $\text{TiS}_2$  powders, Para-(dimethylamino) benzaldehyde ( $\text{C}_9\text{H}_{11}\text{NO}$ ), sodium nitroferricyanide (III) dihydrate ( $\text{Na}_2\text{Fe}(\text{CN})_5\text{NO}\cdot 2\text{H}_2\text{O}$ ), hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and Nafion (5 wt%) were purchased from Aladdin Ltd. (Shanghai, China). Isopropyl alcohol (IPA), Sulfuric acid ( $\text{H}_2\text{SO}_4$ ), ammonium chloride ( $\text{NH}_4\text{Cl}$ ), hydrazine hydrate ( $\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$ ), sodium hypochlorite ( $\text{NaClO}$ ), sodium hydroxide ( $\text{NaOH}$ ), sodium salicylate ( $\text{C}_7\text{H}_5\text{O}_3\text{Na}$ ), sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), hydrochloric acid ( $\text{HCl}$ ), ethanol ( $\text{CH}_3\text{CH}_2\text{OH}$ ) and carbon paper were bought from Beijing Chemical Corporation. The water used throughout all experiments was purified through a Millipore system.

**Preparation of  $\text{TiS}_2$  NSs/CP electrode:** Bulk  $\text{TiS}_2$  powders were added to IPA (10 mL in a 14 mL vial) with an initial concentration of 7.5 mg/mL. These were sonicated using the point probe (sonic tip) for 90 minutes, with a nominal power output of 285 W (38% $\times$ 750 W). After sonication, the dispersions were allowed to settle for 24 hours before centrifuging them at 1500 rpm for 45 minutes. The top 3/4 of the dispersion was collected by pipette. Next, 40  $\mu\text{L}$  of 5 wt% Nafion was added into 960  $\mu\text{L}$  of the obtained solution and sonicated for 1 h to form a homogeneous ink. Then, 140  $\mu\text{L}$  of the dispersion was loaded onto a carbon paper electrode with area of  $1 \times 1 \text{ cm}^2$  and dried under ambient conditions, the catalyst loading mass is  $0.1 \text{ mg cm}^{-2}$ .

**Characterizations:** XRD data were recorded using a Shimadzu XRD-6100 diffractometer with Cu  $\text{K}\alpha$  radiation (40 kV, 30 mA) of wavelength 0.154 nm (SHIMADZU, Japan). SEM images were obtained from a tungsten lamp-equipped SU3500 scanning electron microscope at an accelerating voltage of 20 kV (HITACHI, Japan). TEM images were collected from a HITACHI H-8100 electron microscopy (Hitachi, Tokyo, Japan) operated at 200kV. XPS data were acquired on an ESCALABMK II X-ray photoelectron spectrometer using Mg as the exciting source. The absorbance data of spectrophotometer were measured on SHIMADZU UV-1800 ultraviolet-visible (UV-Vis) spectrophotometer. A gas

chromatography (SHIMADZU, GC-2014C) equipped with MolSieve 5A column and Ar carrier gas was used for H<sub>2</sub> quantifications. Gas-phase product was sampled every 600 s using a gas-tight syringe (Hamilton). The ion chromatography data were collected on 930 Compact IC Flex (Metrohm, Switzerland). The ion chromatography data were collected on Thermofisher ICS 5000 plus using the dual temperature heater, injection valve, conductivity detector, AERS 500 Anions suppressor.

**Electrocatalytic N<sub>2</sub> reduction measurements:** The N<sub>2</sub> reduction experiments were carried out in a two-compartment cell under ambient condition, which was separated by Nafion 117 membrane. The membrane was treated in H<sub>2</sub>O<sub>2</sub> (5%) aqueous solution at 80 °C for 1 h and dipped in 0.1 M H<sub>2</sub>SO<sub>4</sub> at 80°C for another 1 h. Finally, the membrane was treated in ultrapure water at 80°C overnight. The electrochemical experiments were performed with a CHI 660E electrochemical analyzer using a three-electrode configuration with TiS<sub>2</sub> NS/CP electrode, graphite rod and Ag/AgCl electrode (saturated KCl electrolyte) as working electrode, counter electrode and reference electrode, respectively. In all measurements, saturated Ag/AgCl electrode was calibrated with respect to reversible hydrogen electrode as following:  $E \text{ (vs. RHE)} = E \text{ (vs. Ag/AgCl)} + 0.059 \times \text{pH} + 0.197 \text{ V}$ . The presented current density was normalized to the geometric surface area. For electrochemical N<sub>2</sub> reduction, chronoamperometry tests were conducted in N<sub>2</sub>-saturated 0.1 M Na<sub>2</sub>SO<sub>4</sub> solution (30mL). All experiments were carried out under room temperature.

**Determination of NH<sub>3</sub>:** The produced NH<sub>3</sub> was detected with indophenol blue by ultraviolet spectroscopy.<sup>1</sup> In detail, 4 mL electrolyte was obtained from the cathodic chamber and mixed with 50 μL oxidizing solution containing NaClO (4.5%) and NaOH (0.75 M), 500 μL coloring solution containing 0.4 M C<sub>7</sub>H<sub>6</sub>NaO<sub>3</sub> and 0.32 M NaOH, and 50 μL catalyst solution (1 wt % Na<sub>2</sub>Fe(CN)<sub>5</sub>NO·2H<sub>2</sub>O) for 1 h in dark. Absorbance measurements were performed at  $\lambda = 660 \text{ nm}$ . The concentration absorbance curve was calibrated using standard NH<sub>4</sub><sup>+</sup> solution with a series of

concentrations. The fitting curve ( $y = 0.510x + 0.011$ ,  $R^2 = 0.998$ ) shows good linear relation of absorbance value with  $\text{NH}_4^+$  concentration.

**Determination of  $\text{N}_2\text{H}_4$ :** The  $\text{N}_2\text{H}_4$  present in the electrolyte was determined by the method of Watt and Chrisp.<sup>2</sup> The mixture of  $\text{C}_9\text{H}_{11}\text{NO}$  (5.99 g),  $\text{HCl}$  (concentrated, 30 mL), and  $\text{C}_2\text{H}_5\text{OH}$  (300 mL) was used as a color reagent. In detail, 5 mL electrolyte was removed from the electrochemical reaction vessel, and added into 5 mL above prepared color reagent for 10 min at room temperature. Moreover, the absorbance of the resulting solution was measured at a wavelength of 455 nm. The concentration absorbance curves were calibrated using standard  $\text{N}_2\text{H}_4$  solution with a series of concentrations. The fitting curve ( $y = 0.719x + 0.051$ ,  $R^2 = 0.999$ ) shows good linear relation of absorbance value with  $\text{N}_2\text{H}_4$  concentration.

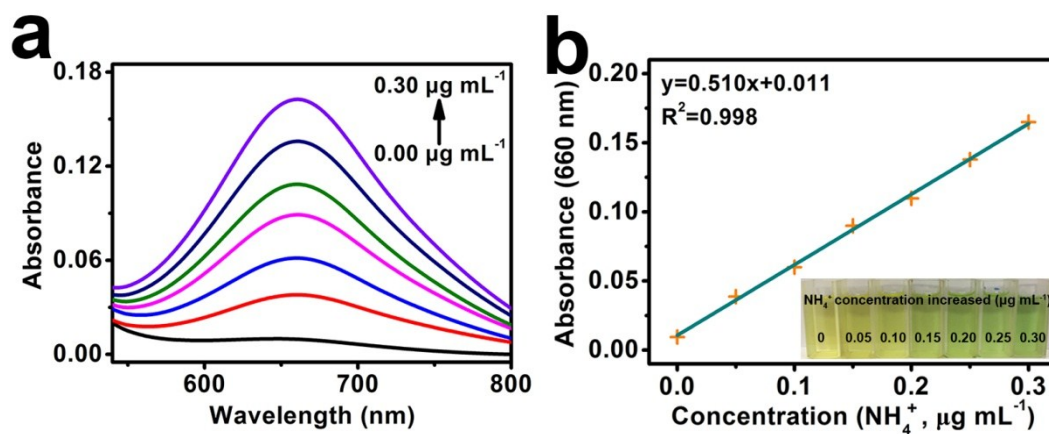
**Determination of FE and  $V_{\text{NH}_3}$ :** The Faradaic efficiency (FE) for  $\text{N}_2$  reduction was defined as the amount of electric charge used for synthesizing  $\text{NH}_3$  divided the total charge passed through the electrodes during the electrolysis. The total amount of  $\text{NH}_3$  produced was measured using colorimetric methods. Assuming three electrons were needed to produce one  $\text{NH}_3$  molecule, the FE could be calculated as follows:

$$\text{FE} = \frac{3F \times [\text{NH}_4^+] \times V}{18 \times Q}$$

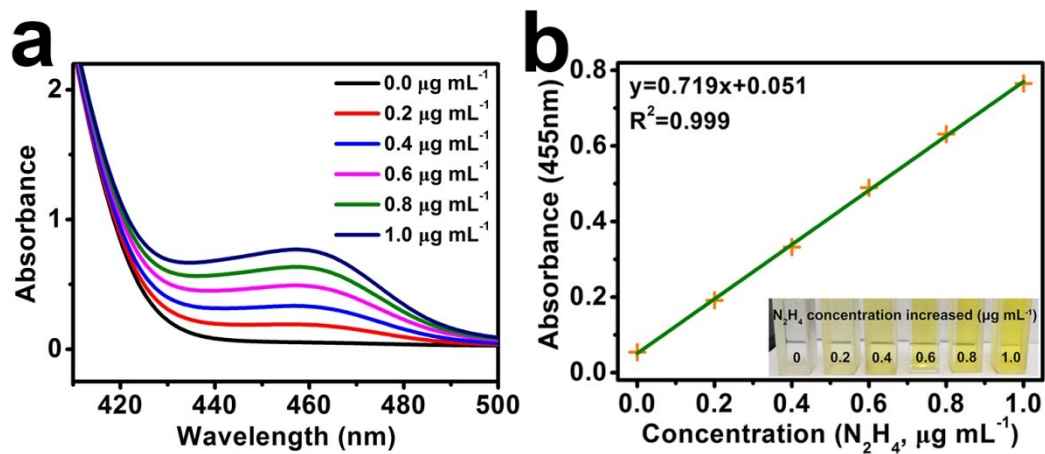
The rate of  $\text{NH}_3$  formation was calculated using the following equation:

$$V_{\text{NH}_3} = \frac{[\text{NH}_4^+] \times V}{t \times m_{\text{cat.}}}$$

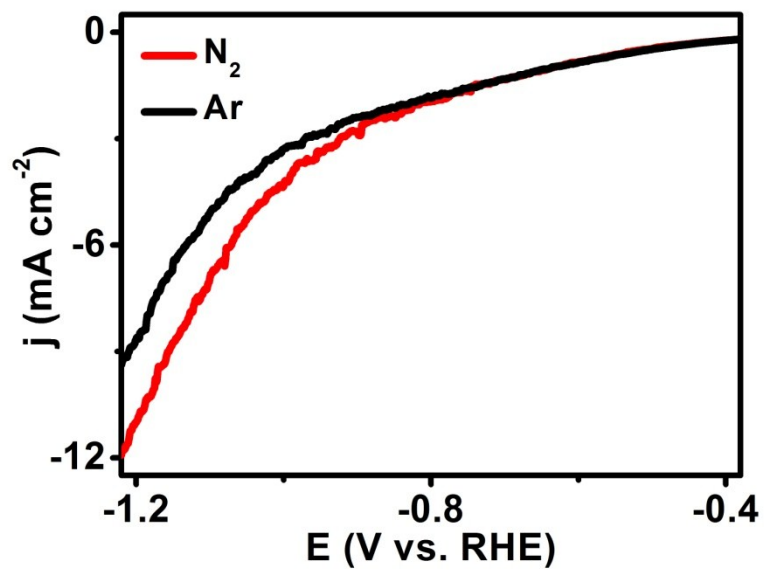
Where  $F$  is the Faraday constant,  $[\text{NH}_4^+]$  is the measured  $\text{NH}_4^+$  concentration,  $V$  is the volume of the electrolyte in the cathodic chamber,  $Q$  is the total quantity of applied electricity;  $t$  is the reduction time;  $m_{\text{cat.}}$  is the loaded mass of catalyst on carbon paper.



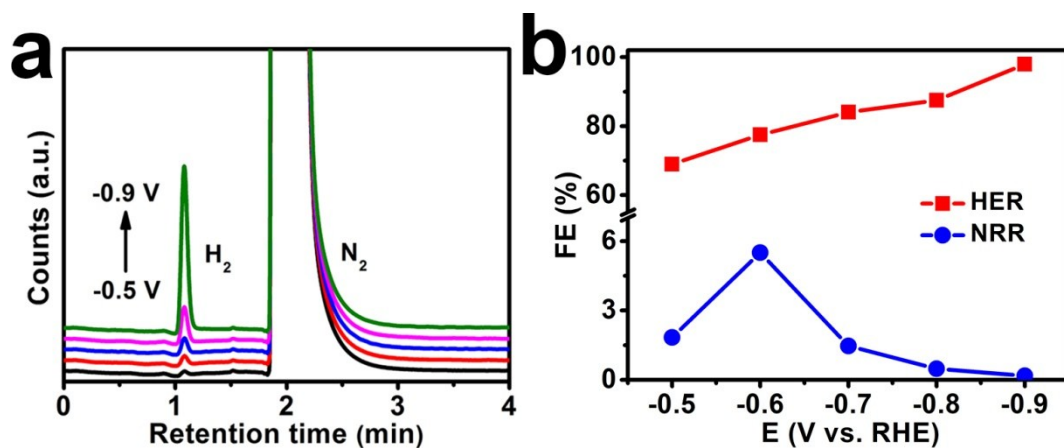
**Fig. S1.** (a) UV-vis absorption spectra of various  $\text{NH}_4^+$  concentrations after incubated for 2 hours at room temperature. (b) Calibration curve used for calculation of  $\text{NH}_4^+$  concentrations.



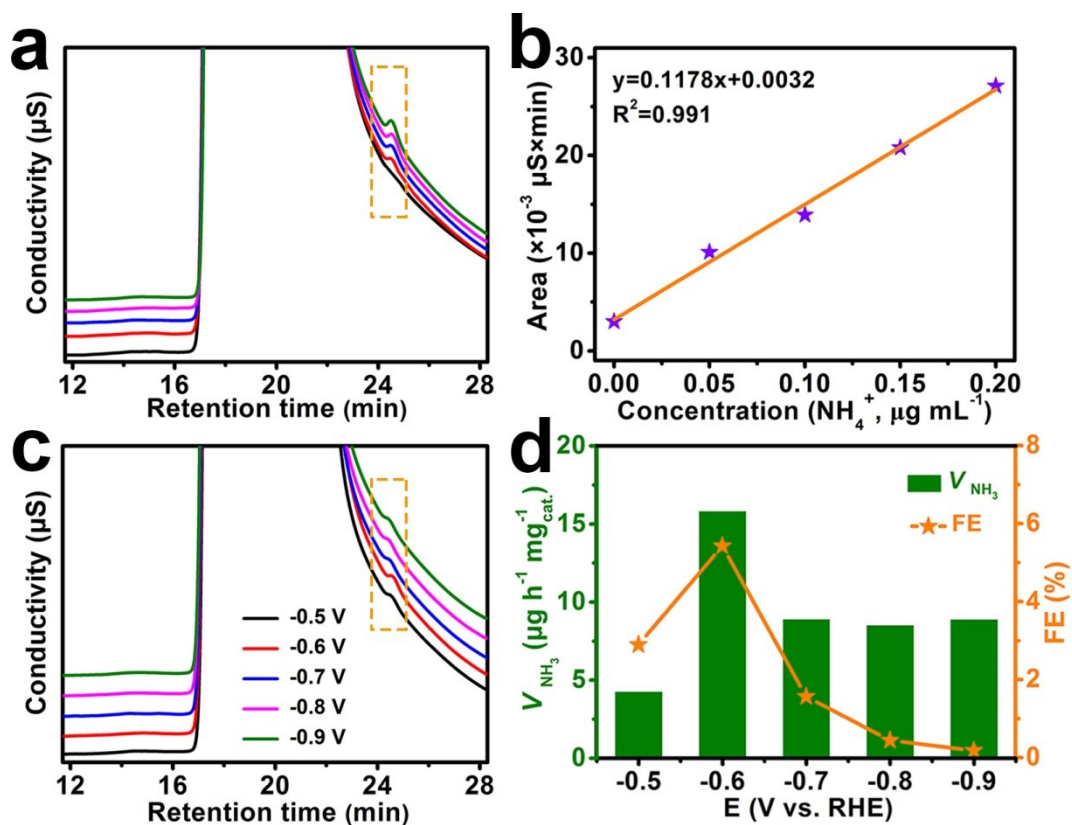
**Fig. S2.** (a) UV–vis absorption spectra of various  $N_2H_4$  concentrations after incubated for 20 min at room temperature. (b) Calibration curve used for calculation of  $N_2H_4$  concentrations.



**Fig. S3.** LSV curves of TiS<sub>2</sub> NSs/CP in Ar- and N<sub>2</sub>-saturated 0.1 M Na<sub>2</sub>SO<sub>4</sub> with a scan rate of 2 mV s<sup>-1</sup>.

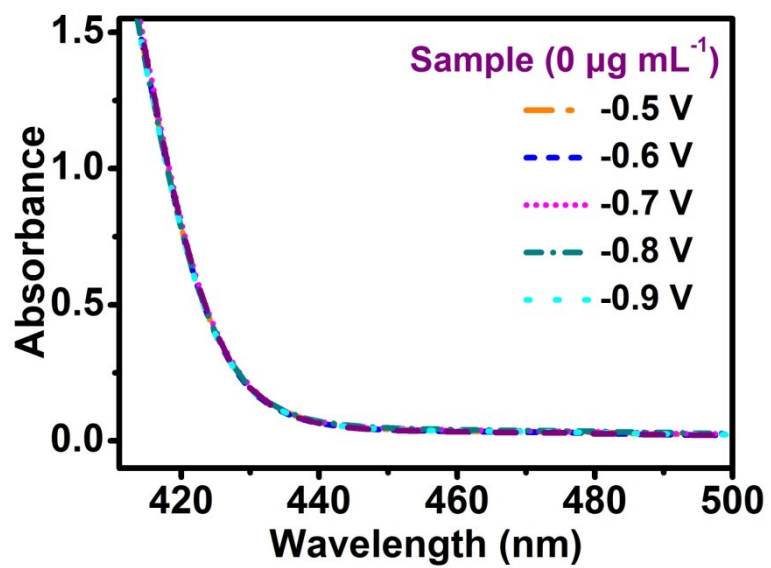


**Fig. S4.** (a) gas chromatography (GC) spectra of the gas from on-line measurements collected by GC for the NRR on TiS<sub>2</sub> NSs/CP in N<sub>2</sub>-saturated 0.1 M Na<sub>2</sub>SO<sub>4</sub> at various potentials. The on-line measurements were collected every 10 minutes. (b) The calculated FEs of HER and NRR at various potentials.

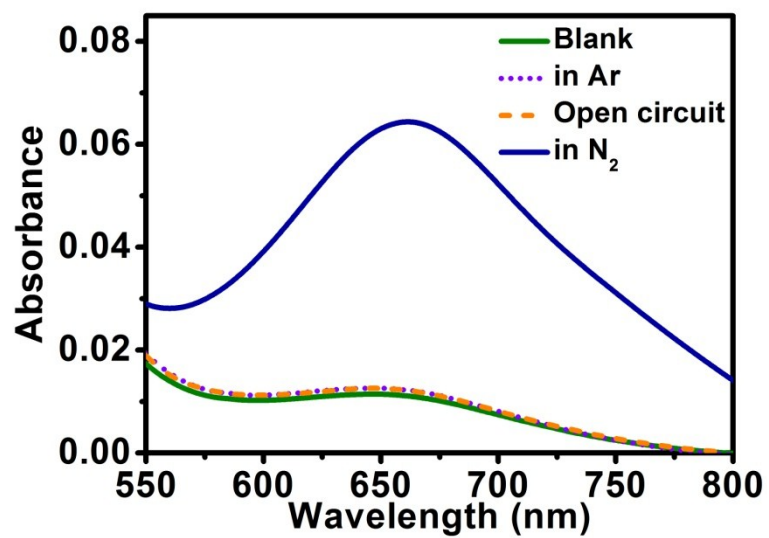


**Fig. S5.** (a) Ion chromatogram analysis for the  $\text{NH}_4^+$  ions. (b) Calibration curve used for estimation of  $\text{NH}_4^+$ . (c) Ion chromatogram for the electrolytes at a series of potentials after electrolysis for 2 h. (d)  $\text{NH}_3$  yields and FEs for  $\text{TiS}_2$  NSs/CP at corresponding potentials.

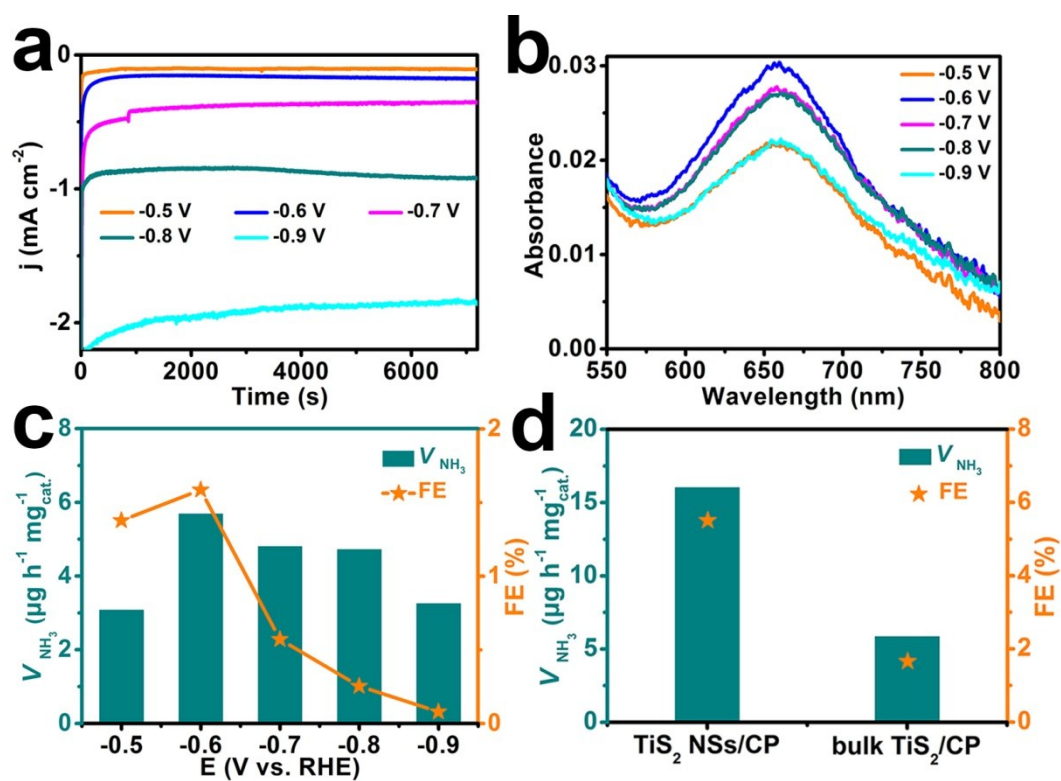




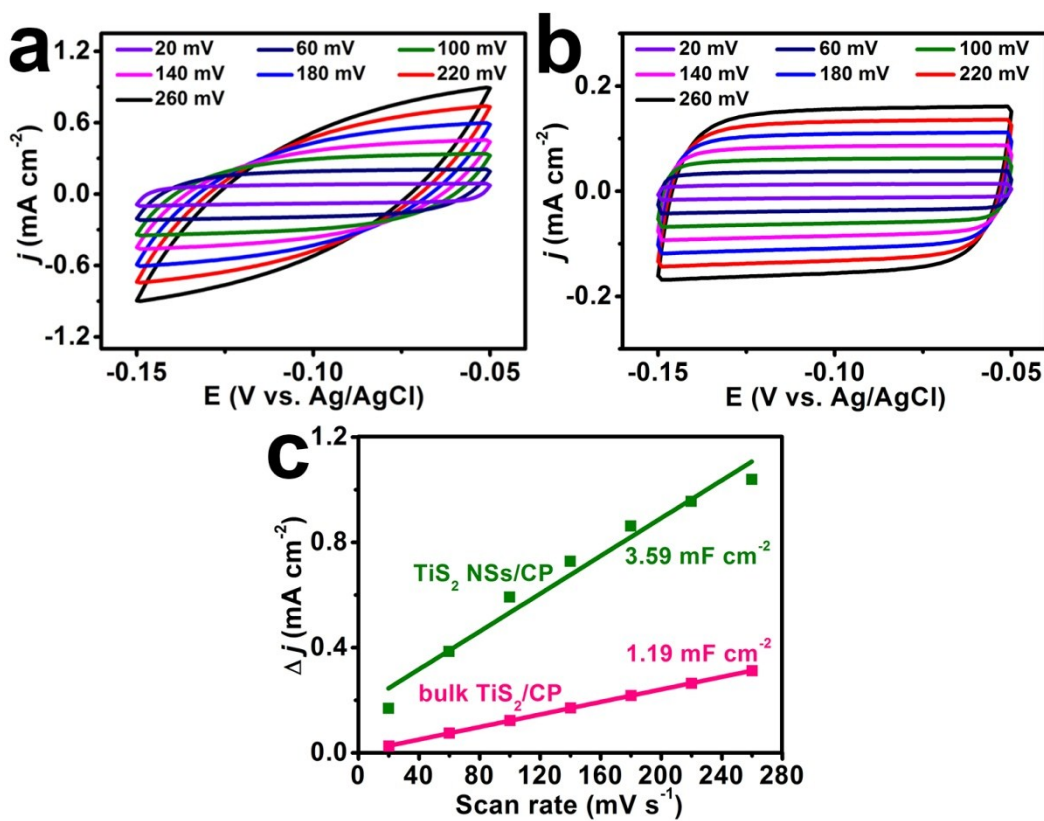
**Fig. S6.** UV-vis spectra of the electrolyte estimated by the method of Watt and Chrisp before and after 2 h electrolysis in N<sub>2</sub> atmosphere at a series of potentials under ambient conditions.



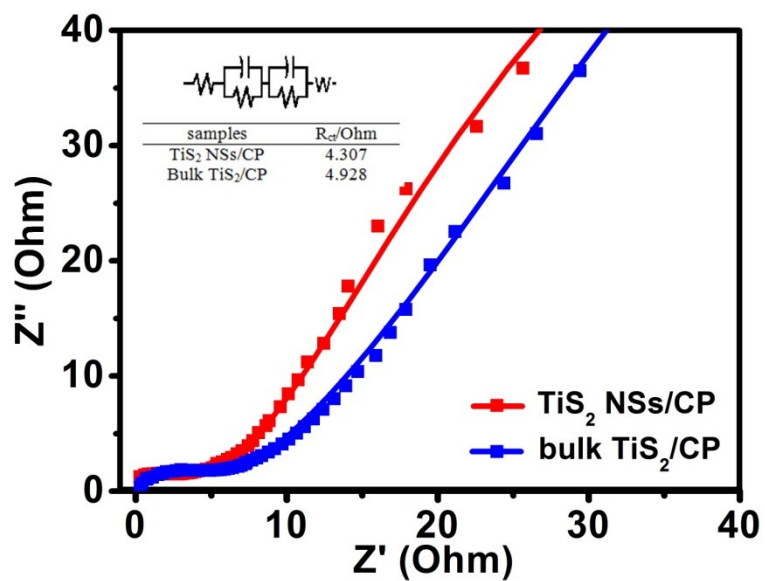
**Fig. S7.** UV-vis absorption spectra of the electrolyte stained with indophenol indicator after charging at  $-0.6$  V for 2 h under different electrochemical conditions.



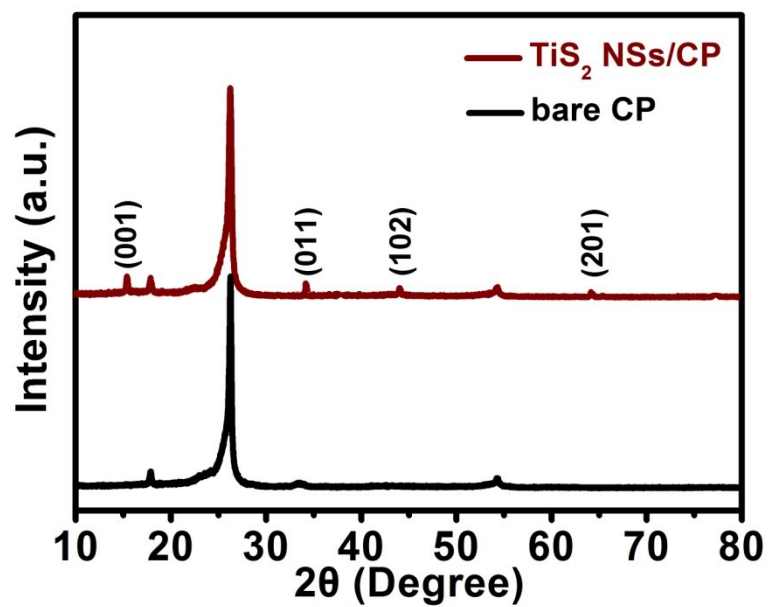
**Fig. S8.** (a) Time-dependent current density curves for bulk  $\text{TiS}_2/\text{CP}$  at different potentials in  $\text{N}_2$ -saturated 0.1 M  $\text{Na}_2\text{SO}_4$ . (b) UV-vis absorption spectra of the electrolytes stained with indophenol indicator after electrolysis at a series of potentials for 2 h. (c)  $\text{NH}_3$  yields and FEs of bulk  $\text{TiS}_2/\text{CP}$  for NRR at a series of potentials. (d)  $\text{NH}_3$  yields and FEs with different electrodes at -0.6 V after 2 h electrolysis under ambient conditions.



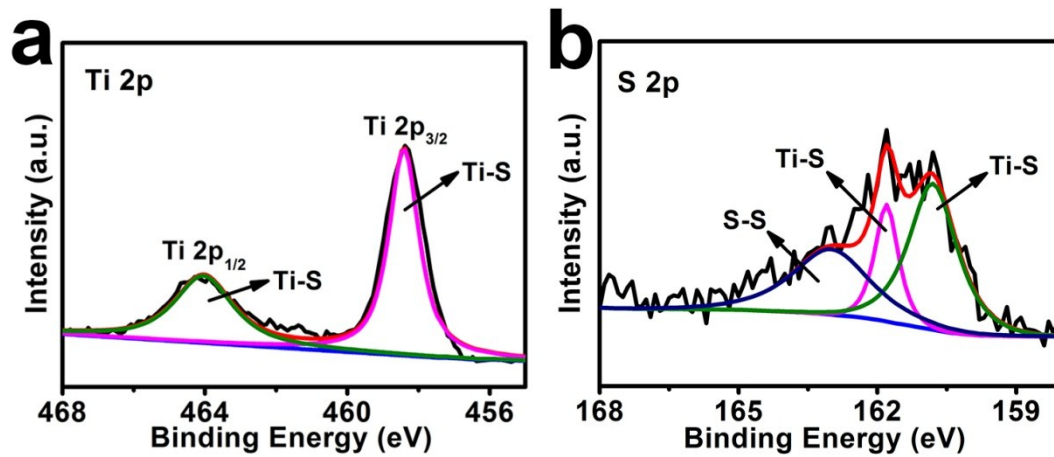
**Fig. S9.** Cyclic voltammetry curves of (a) TiS<sub>2</sub> NSs/CP and (b) bulk TiS<sub>2</sub>/CP with various scan rates (20-260 mV s<sup>-1</sup>) in the region of -0.05 to -0.15 V vs. Ag/AgCl. (c) The capacitive current densities at -0.10 V vs. Ag/AgCl as a function of scan rates for TiS<sub>2</sub> NSs/CP and bulk TiS<sub>2</sub>/CP.



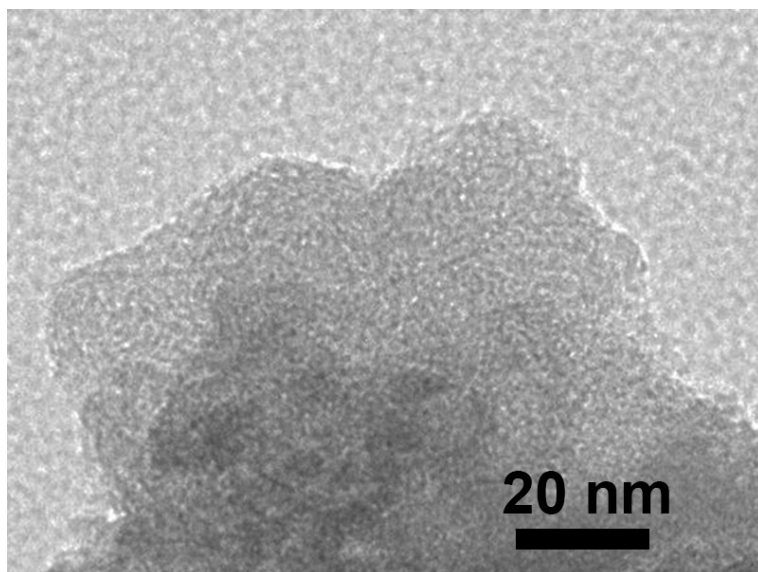
**Fig. S10.** Nyquist plots and fitting lines of TiS<sub>2</sub> NSs/CP and bulk TiS<sub>2</sub>/CP with a voltage amplitude of 5 mV, and all three electrodes are in one compartment cell being full of 0.1 M Na<sub>2</sub>SO<sub>4</sub> solution.



**Fig. S11.** XRD patterns for TiS<sub>2</sub> NSs/CP and bare CP after long-term electrocatalysis in 0.1 M Na<sub>2</sub>SO<sub>4</sub>.



**Fig. S12.** XPS spectra for TiS<sub>2</sub> NSs/CP in the (a) Ti 2p, (b) S 2p regions after long-term electrocatalysis in 0.1 M Na<sub>2</sub>SO<sub>4</sub>.



**Fig. S13.** TEM image for TiS<sub>2</sub> NSs after long-term electrocatalysis in 0.1 M Na<sub>2</sub>SO<sub>4</sub>.



**Table S1.** Comparison of ambient N<sub>2</sub> reduction performance for TiS<sub>2</sub> nanosheets with other aqueous-based NRR electrocatalysts.

Catalyst	Electrolyte	NH <sub>3</sub> yield	FE (%)	Ref.
TiS <sub>2</sub> NSs	0.1 M Na <sub>2</sub> SO <sub>4</sub>	16.02 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	5.50	This work
MoS <sub>2</sub> /CC	0.1 M Na <sub>2</sub> SO <sub>4</sub>	8.08 × 10 <sup>-11</sup> mol s <sup>-1</sup> cm <sup>-2</sup>	1.17	3
TiO <sub>2</sub>	0.1 M Na <sub>2</sub> SO <sub>4</sub>	9.16 × 10 <sup>-11</sup> mol s <sup>-1</sup> cm <sup>-2</sup>	2.5	4
TiO <sub>2</sub> -rGO	0.1 M Na <sub>2</sub> SO <sub>4</sub>	15.13 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	3.3	5
C-TiO <sub>2</sub>	0.1 M Na <sub>2</sub> SO <sub>4</sub>	16.22 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	1.84	6
B-TiO <sub>2</sub>	0.1 M Na <sub>2</sub> SO <sub>4</sub>	14.4 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	3.4	7
defect-rich MoS <sub>2</sub> nanoflower	0.1 M Na <sub>2</sub> SO <sub>4</sub>	29.28 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	8.34	8
d-TiO <sub>2</sub> /TM	0.1 M HCl	1.24 × 10 <sup>-10</sup> mol s <sup>-1</sup> cm <sup>-2</sup>	9.17	9
N-doped porous carbon	0.05 M H <sub>2</sub> SO <sub>4</sub>	23.8 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	1.42	10
Mo nanofilm	0.01 M H <sub>2</sub> SO <sub>4</sub>	3.09 × 10 <sup>-11</sup> mol s <sup>-1</sup> cm <sup>-2</sup>	0.72	11
γ-Fe <sub>2</sub> O <sub>3</sub>	0.1 M KOH	0.212 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	1.9	12
Pd <sub>0.2</sub> Cu <sub>0.8</sub> /rGO	0.1 M KOH	1.66 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	4.5	13
Fe <sub>2</sub> O <sub>3</sub> nanorods	0.1 M Na <sub>2</sub> SO <sub>4</sub>	15.9 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	0.94	14
PEBCD/C	0.5 M Li <sub>2</sub> SO <sub>4</sub>	1.58 μg h <sup>-1</sup> cm <sup>-2</sup>	2.85	15
Fe <sub>2</sub> O <sub>3</sub> -CNT	KHCO <sub>3</sub>	0.22 μg h <sup>-1</sup> cm <sup>-2</sup>	0.15	16
Au nanorods	0.1 M KOH	6.042 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	4.0	17
TA-reduced Au/TiO <sub>2</sub>	0.1 M HCl	21.4 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	8.11	18
α-Au/CeO <sub>x</sub> -RGO	0.1 M HCl	8.31 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	10.10	19
MoN	0.1 M HCl	3.01 × 10 <sup>-10</sup> mol s <sup>-1</sup> cm <sup>-2</sup>	1.15	20
Ru/C	2.0 M KOH	0.25 μg h <sup>-1</sup> cm <sup>-2</sup>	0.92	21
Fe <sub>3</sub> O <sub>4</sub> /Ti	0.1 M Na <sub>2</sub> SO <sub>4</sub>	5.6 × 10 <sup>-11</sup> mol s <sup>-1</sup> cm <sup>-2</sup>	2.6	22
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	23
MoO <sub>3</sub>	0.1 M HCl	29.43 μg h <sup>-1</sup> mg <sup>-1</sup> <sub>cat.</sub>	1.9	24

VN	0.1 M HCl	$8.40 \times 10^{-11} \text{ mol s}^{-1} \text{ cm}^{-2}$	2.25	25
Nb <sub>2</sub> O <sub>5</sub> nanofiber	0.1 M HCl	$43.6 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	9.26	26
Mn <sub>3</sub> O <sub>4</sub> nanocube	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$11.6 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	3.0	27
Ti <sub>3</sub> C <sub>2</sub> T <sub>x</sub> nanosheet	0.1 M HCl	$20.4 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	9.3	28
MnO	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$1.11 \times 10^{-10} \text{ mol s}^{-1} \text{ cm}^{-2}$	8.02	29
AuSAs-NDPCs	0.1 M HCl	$2.32 \mu\text{g h}^{-1} \text{ cm}^{-2}$	12.3	30
AuHNCs	0.5 M LiClO <sub>4</sub>	$3.98 \mu\text{g h}^{-1} \text{ cm}^{-2}$	14.8	31
B <sub>4</sub> C	0.1 M HCl	$26.57 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	15.95	32
hollow Cr <sub>2</sub> O <sub>3</sub> microspheres	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$25.3 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	6.78	33
Ag nanosheets	0.1 M HCl	$4.62 \times 10^{-11} \text{ mol s}^{-1} \text{ cm}^{-2}$	4.8	34
$\beta$ -FeOOH nanorods	0.5 M LiClO <sub>4</sub>	$23.32 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	6.7	35
SnO <sub>2</sub>	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$1.47 \times 10^{-10} \text{ mol s}^{-1} \text{ cm}^{-2}$	2.17	36
porous bromide-derived Ag film	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$2.07 \times 10^{-11} \text{ mol s}^{-1} \text{ cm}^{-2}$	7.36	37
S-doped carbon nanospheres	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$19.07 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	7.47	38
oxygen-doped carbon nanosheet	0.1 M HCl	$20.15 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	4.97	39
Nb <sub>2</sub> O <sub>5</sub> nanowires array/CC	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$1.58 \times 10^{-10} \text{ mol s}^{-1} \text{ cm}^{-2}$	2.26	40
VO <sub>2</sub> hollow microsphere	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$14.85 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	3.97	41
Ru SAs/N-C	0.05 M H <sub>2</sub> SO <sub>4</sub>	$120.9 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	29.6	42
Au flowers	0.1 M HCl	$25.57 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	6.05	43
NbO <sub>2</sub> nanoparticles	0.05 M H <sub>2</sub> SO <sub>4</sub>	$11.6 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	19.7	44
Fe <sub>3</sub> S <sub>4</sub> nanosheets	0.1 M HCl	$75.4 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	6.45	45
La <sub>2</sub> O <sub>3</sub>	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$17.04 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	4.76	46
CoO quantum dots	0.1 M Na <sub>2</sub> SO <sub>4</sub>	$21.5 \mu\text{g h}^{-1} \text{ mg}^{-1}_{\text{cat.}}$	8.3	47
Bi nanosheet array	0.1 M HCl	$6.89 \times 10^{-11} \text{ mol s}^{-1} \text{ cm}^{-2}$	10.26	48

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