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Surface engineering of ReS_2 by cobalt for efficient performance of hydrogen evolution under both acid and alkaline conditions

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Synthesis of ReS_2/Co-x

ReS_2/Co-x **electrocatalysts** (x = 0, 1%, 2%, 5%, 8%) were synthesized via one-step hydrothermal approach (Fig. 1). Firstly, 1 mmol $NaReO_4$ and a certain amount of $Na_2S{\cdot}9H_2O$ were added into 74 ml deionized (DI) water with vigorous stirring. Next, 2 mmol hydroxylamine hydrochloride as well as a proportional amount of $Co(NO_3)_2{\cdot}6H_2O$ were dropped into above solution, respectively. Then, transferred solution into Teflon-lined stainless steel autoclave at the temperature of 200 °C for one day. Finally, washed the products repeatedly via DI water and ethanol. The fabricated ReS_2/Co-x **electrocatalysts** (x =0, 1%, 2%, 5%, 8%) with different ratio were denoted as RC-x. **The content of ReS_2/Co-x electrocatalysts are detected by using Inducitively coupled plasma and the results are listed in Table S1.**

Characterization

X-ray diffraction (XRD) characterization was employed to analyze catalyst structure by a Shimadzu/XD-3A diffractometer system. Copper Kα radiation (λ = 1.5418 Å) was used. Morphologies of the catalysts were employed through transmission electron microscopy (TEM) by using an JEOL 2100 system. X-ray photoelectron spectra (XPS) was carried out by using PHI 5000 Versaprobe with Al-Kα radiation. Electron spin-resonance spectroscopy (ESR) was performed by Jes FA200. **Raman was employed by using Bruker senterra. Inducitively coupled plasma was used by a Agilent 7700 equipment.**

Electrochemical measurements

The synthesized catalysts was tested in 0.5M H_2SO_4 (purged with pure N_2) using a typical three electrode setup on an electrochemical station (Chenhua Instruments, CHI660D) with a Ag/AgCl reference electrode, a graphite rod as counter electrode and a glassy carbon electrode (GCE) covered with ReS_2 composites as working electrode to study the electrochemical property. All potential data are given versus reversible hydrogen electrode (RHE) according to the following equation: $E_{RHE} = E_{Ag/AgCl} + 0.197 + 0.059 \times pH\ (V)$. Linear sweep voltammetry (LSV) was conducted in 0.5 M H_2SO_4 with a scan rate of 5 mV s^{-1}. The current density vs potential data plots were corrected for 90% ohmic compensation throughout the system. For a Tafel plot, the linear portion fits the Tafel equation ($\eta = b\log|j| + a$) to achieve the Tafel slope. Cyclic voltammetry (CV) was measured with scan rates of from 0.01 to 0.1 V for the investigations of electrochemical surface areas. The electrochemical impedance spectroscopy (EIS) measurements were carried out with frequencies ranging from 100 kHz to 0.1 Hz. The stability of the catalyst was tested by long time chronopotentiometry at the current density of 10 mA cm^{-2}.

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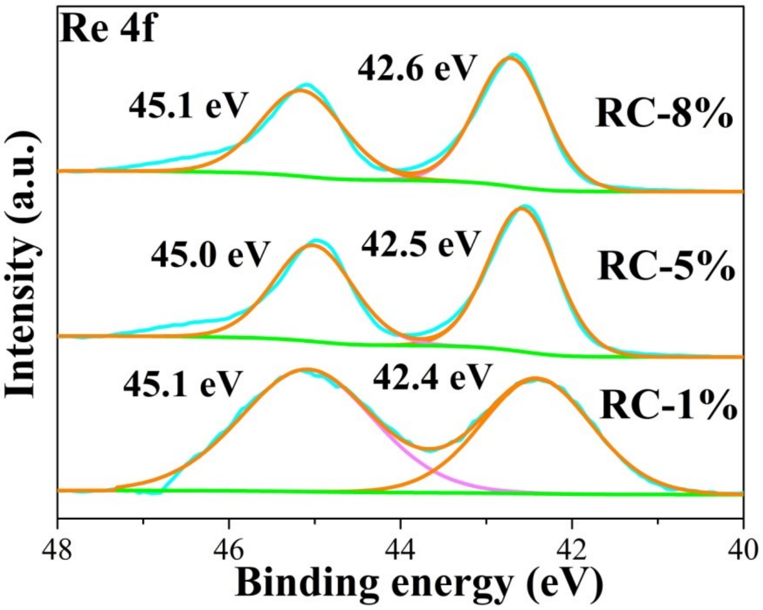

Fig.S1. X-ray photoelectron spectra (XPS) results of Re 4f in RC-1%, RC-5% and RC-8% samples.

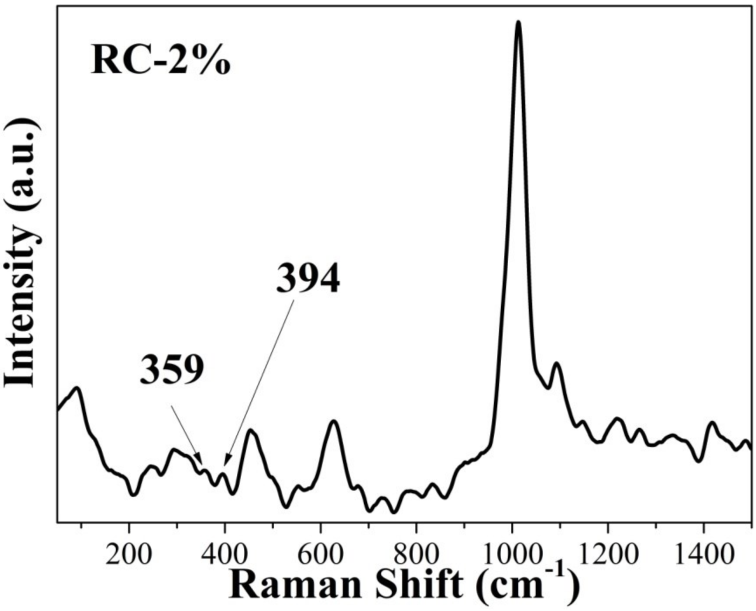

Fig.S2. Raman spectrum of RC-2% sample.

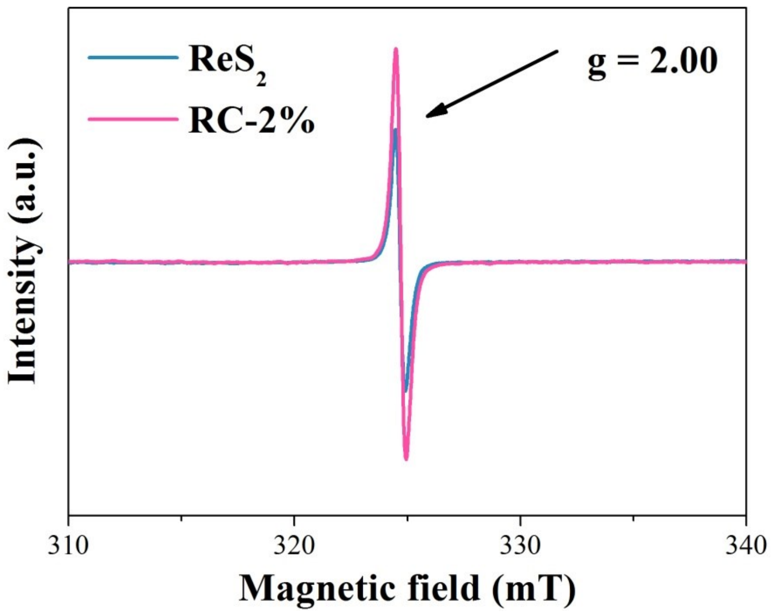

Fig.S3. Electron spin-resonance spectroscopy of RC-2% and ReS$_2$ electrocatalysts.

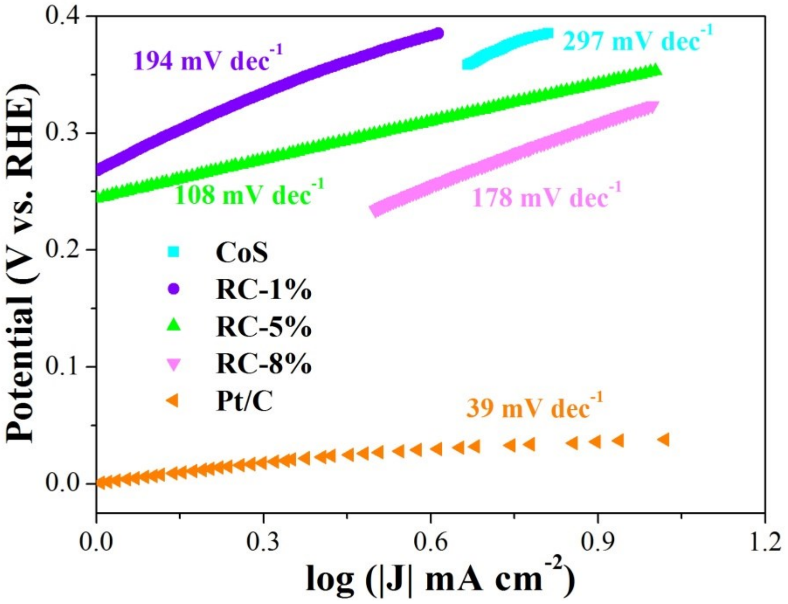

Fig. S4. Tafel plot of CoS, Pt/C and RC catalysts from the corresponding LSV curves in 0.5 M H_2SO_4.

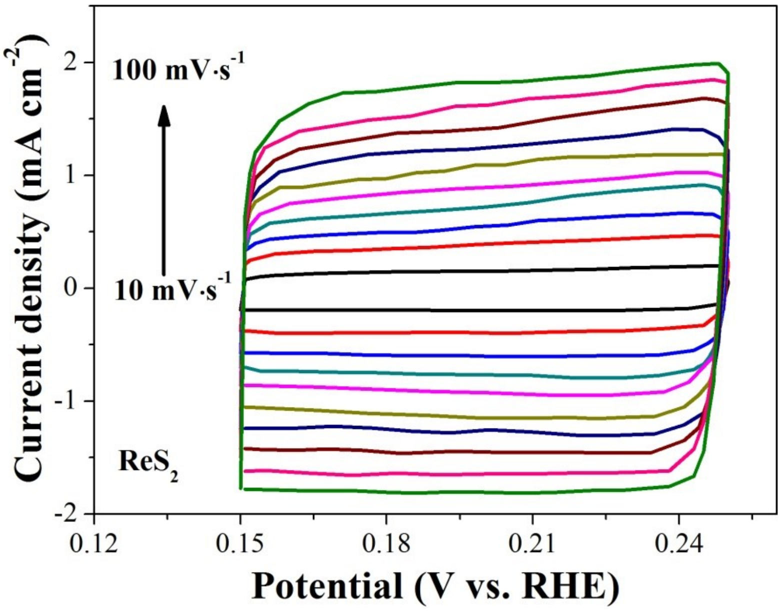

Fig. S5. CVs for pure ReS_2 with different rates from 10 to 100 mV s^{-1} in the potential range of 0.15-0.25 V.

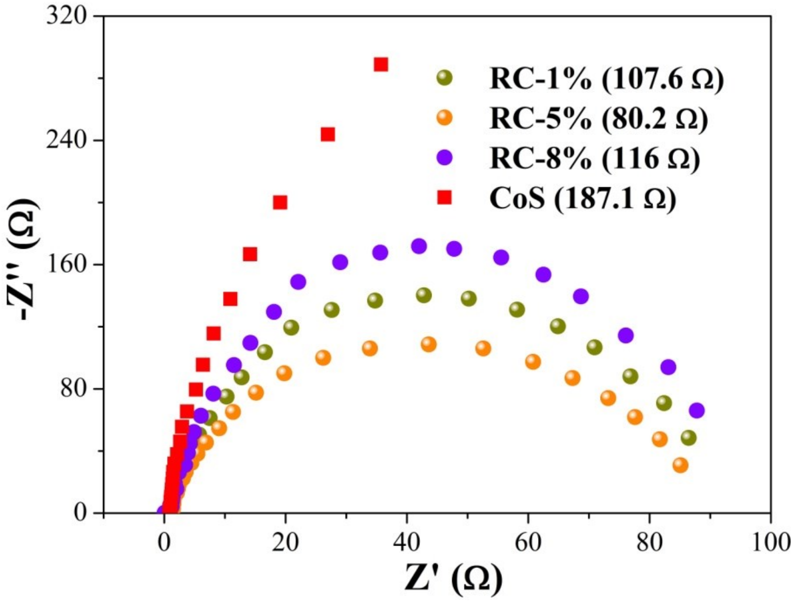

Fig. S6. Electrochemical impedance spectroscopy (EIS) Nyquist plots for pure CoS and RC-x in 0.5 M H_2SO_4.

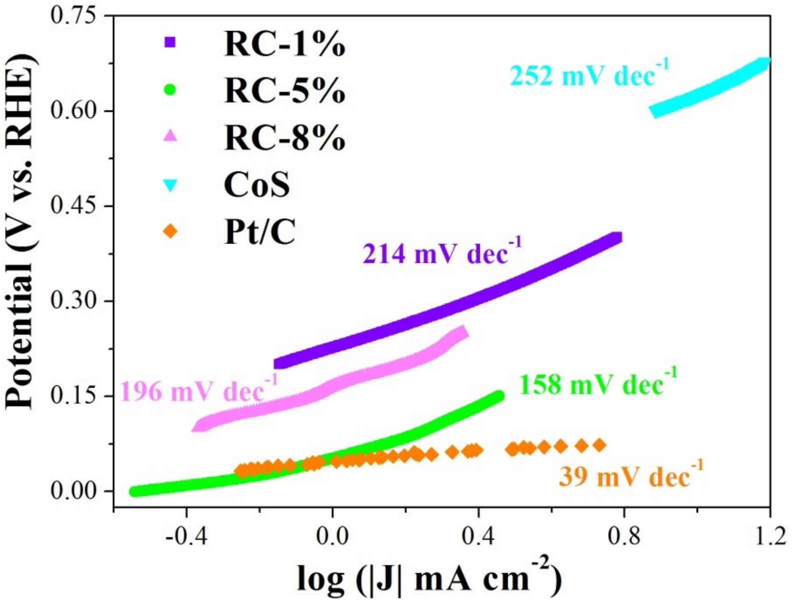

Fig. S7. Tafel plot of CoS, Pt/C and RC catalysts from the corresponding LSV curves in 1 M KOH.

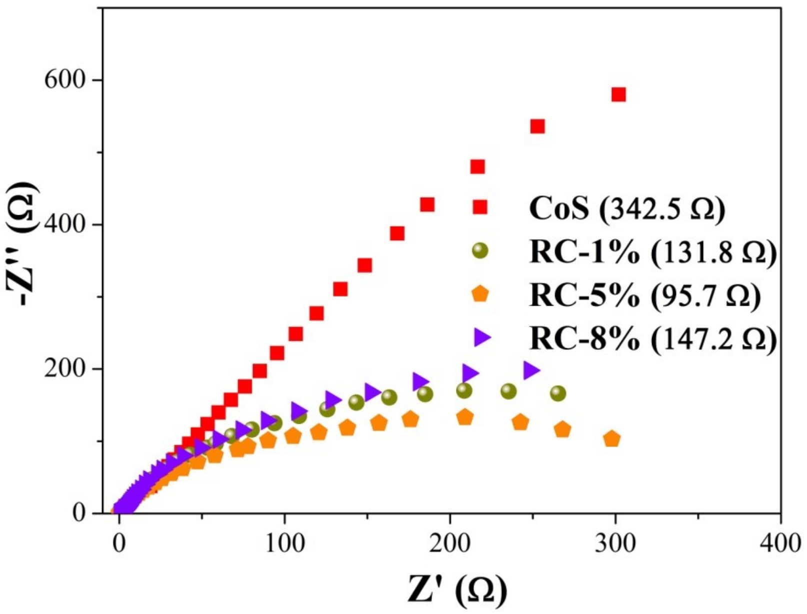

Fig. S8. Electrochemical impedance spectroscopy (EIS) Nyquist plots for pure CoS and RC-x in 1 M KOH.

Table S1. Inducitively coupled plasma results of ReS_2/Co-x.

samples	RC-1%	RC-2%	RC-5%	RC-8%
Co (wt. %)	0.7	1.6	4.0	6.5