

*Supplementary Information*

## **Defect- and S-rich ultrathin MoS<sub>2</sub> nanosheet embedded N-doped carbon nanofibers for efficient hydrogen evolution**

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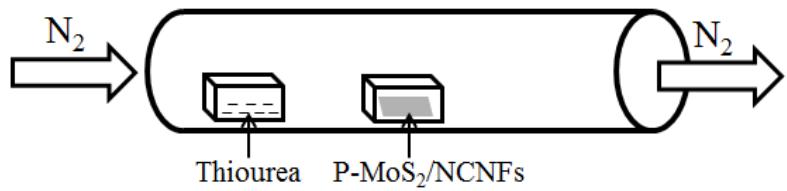


Figure S1. The stabilization and carbonization of MoS<sub>2</sub>/NCNFs.

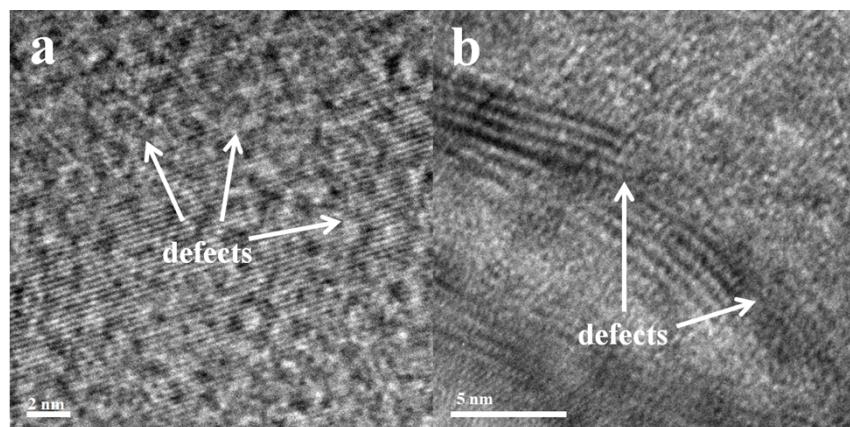


Figure S2. HRTEM images of MoS<sub>2</sub>/NCNFs: (a) interplanar, (b) interlayer.

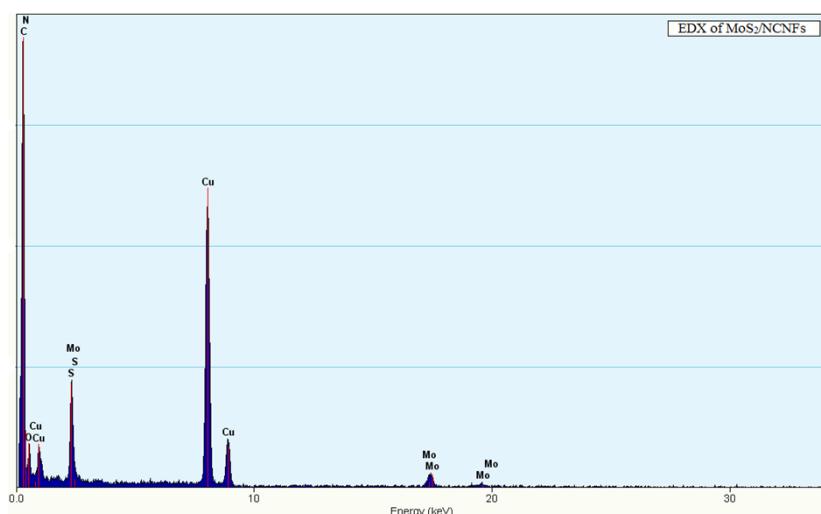


Figure S3. The energy dispersive X-ray (EDX) spectra of MoS<sub>2</sub>/NCNFs

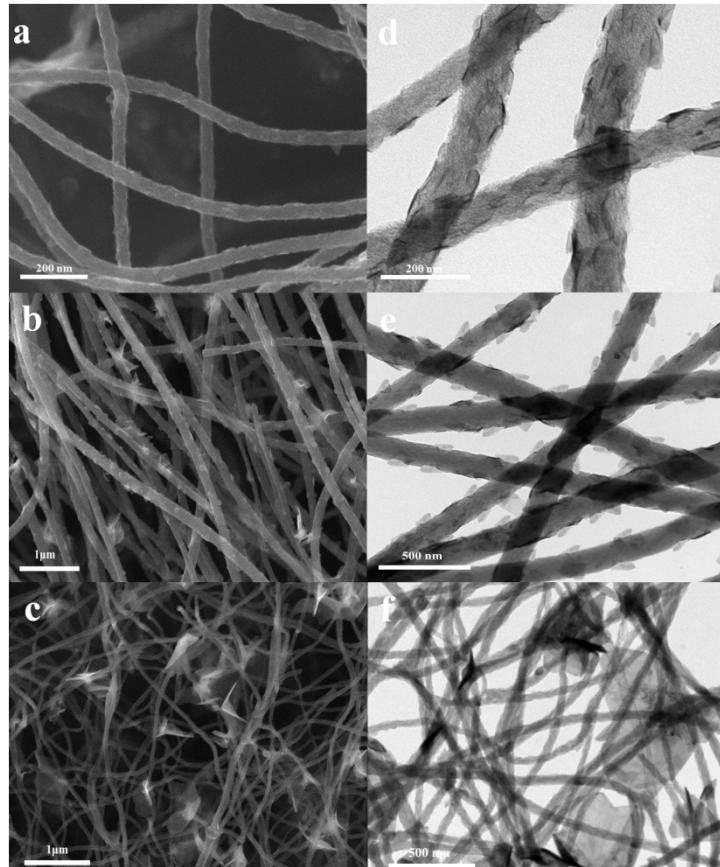


Figure S4. SEM and TEM images of the MoS<sub>2</sub>/NCNFs prepared at different heating rates: (a, d) 5, (b, e) 10 and (c, f) 15 °C min<sup>-1</sup>.

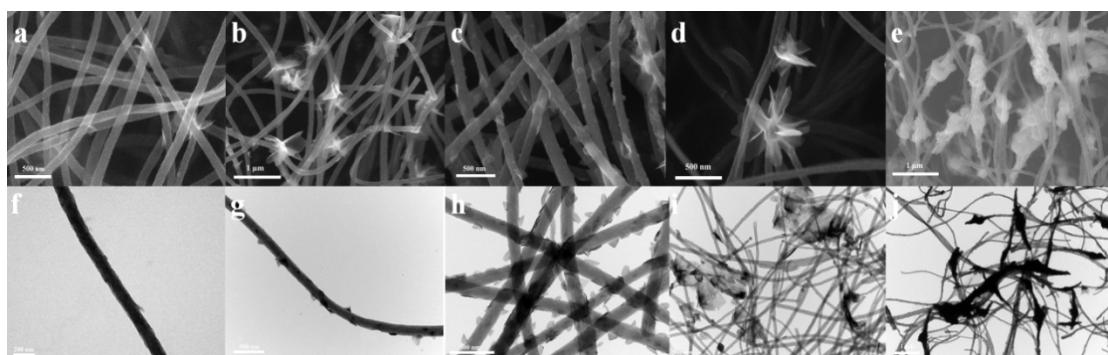


Figure S5. SEM and TEM images of the MoS<sub>2</sub>/NCNFs prepared at different concentration of Mo precursor: (a, f) 1.0 mM, (b, g) 1.5 mM, (c, h) 2.0 mM, (d, i) 2.5 mM and (e, j) 3.0 mM.

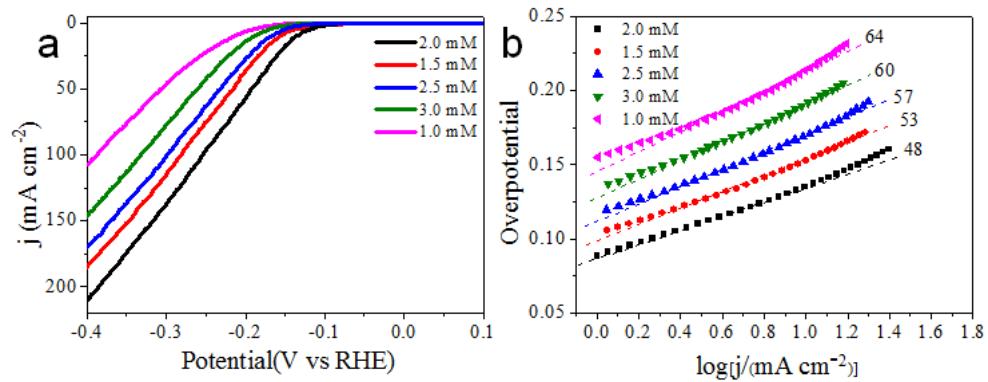


Figure S6. Polarization curves (a) and the corresponding Tafel plots (b) of MoS<sub>2</sub>/NCNFs prepared at different concentration of Mo precursor.

Table S1. Electrochemical parameters of the MoS<sub>2</sub>/NCNFs prepared at different concentration of Mo precursor.

Concentration	Overpotentials at current density of 10 mA cm <sup>-2</sup> (mV)	Current density at η = 200 mV (mA cm <sup>-2</sup> )	Tafel slopes (mV dec <sup>-1</sup> )	J <sub>0</sub> (μA cm <sup>-2</sup> )
1.0 mM	215	6.8	64	4.7
1.5 mM	156	40.4	53	16.3
2.0 mM	135	65.6	48	24.2
2.5 mM	167	28.2	57	10.4
3.0 mM	188	14.7	60	7.0

Table S2. Electrochemical parameters of different MoS<sub>2</sub> catalysts

Catalyst	Mass loading (mg cm <sup>-2</sup> )	Tafel slopes (mV dec <sup>-1</sup> )	Overpotentials at current density of 10 mA cm <sup>-2</sup> (mV)	Current density at η = 200 mV (mA cm <sup>-2</sup> )	Current density at η = 300 mV (mA cm <sup>-2</sup> )	J <sub>0</sub> (μA cm <sup>-2</sup> )
<b>Oxygen-incorporated MoS<sub>2</sub> nanosheets<sup>16</sup></b>	0.285	55	N/A	N/A	126.5	12.6
<b>Defect-rich MoS<sub>2</sub> nanosheets<sup>17</sup></b>	0.285	50	N/A	13	70	8.91
<b>S-rich MoS<sub>2</sub>-NCNFs<sup>37</sup></b>	0.857	38	120	N/A	N/A	N/A
<b>MoS<sub>x</sub>/NCNT<sup>38</sup></b>	0.102	40	110	N/A	N/A	33.11
<b>MoS<sub>2</sub> nanosheets within graphite<sup>44</sup></b>	0.200	41	N/A	23	N/A	N/A
<b>MoS<sub>2</sub> ⊥ RGO<sup>50</sup></b>	0.204	43	172	N/A	N/A	N/A
<b>Defect- and S-rich MoS<sub>2</sub>/NCNFs (current work)</b>	0.217	48	135	65.6	N/A	24.2

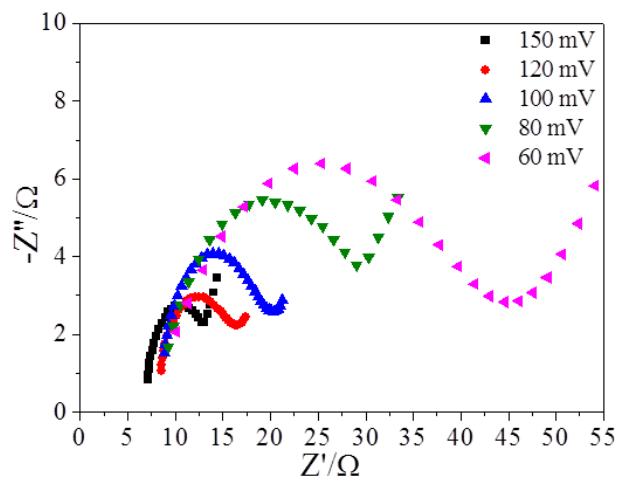


Figure S7. Nyquist plots of  $\text{MoS}_2/\text{NCNFs}$  at various overpotentials of 60 mV, 80 mV, 100 mV, 120 mV and 150 mV.

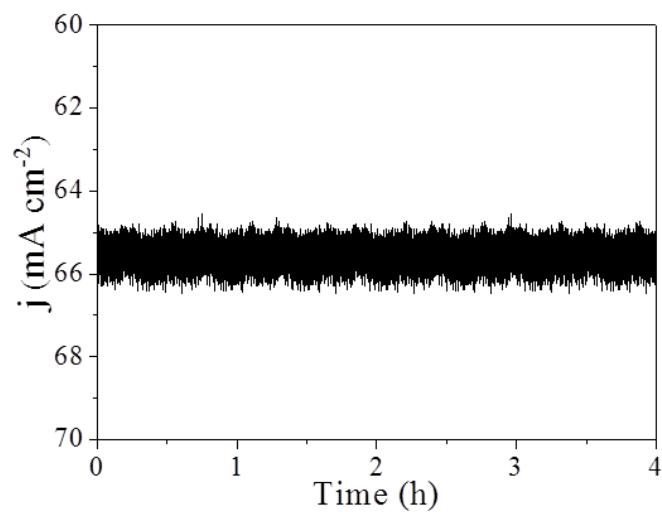


Figure S8. Potentiostatic electrolysis of  $\text{MoS}_2/\text{NCNFs}$  for 4 h. The potential we applied is 200 mV vs RHE after iR correction.