

[Supporting Information]

Stabilized Monolayer 1T MoS₂ Embedded in CoOOH for Highly Efficient Overall Water Splitting

Bo Shang,^a Pengfei Ma,^a Jinchang Fan,^a Lin Jiao,^a Zijian Liu,^a Zheyu Zhang,
^a Nuo Chen,^a Zhiliang Cheng,^b Xiaoqiang Cui*^a and Weitao Zheng*^a

*a. State Key Laboratory of Automotive Simulation and Control, School of
Materials Science and Engineering, Jilin University, 2699 Qianjin Street,
Changchun 130012, People's Republic of China.*

*b. Department of Bioengineering, University of Pennsylvania, 210 South 33rd
Street, 240 Skirkanich Hall, Philadelphia, PA, 19104, USA*

*Corresponding author, E-mail: xqcui@jlu.edu.cn

Experimental Section:

1. *Materials and reagents.* Cobalt acetyl acetone ($\text{Co}(\text{acac})_2$) was purchased from Aladdin reagent, China. Hexaammonium heptamolybdate tetrahydrate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$, AR), N, N-dimethylformamide (DMF, AR), and Thiourea (AR) were purchased from Sinopharm chemical reagent, China. Commercial Pt/C (40 wt. %), IrO_2 , and MoS_2 powder were obtained from Sigma-Aldrich. Sulfuric acid (H_2SO_4 , AR) and ethanol (AR) were obtained from Beijing chemical works, China. Nafion-ethanol solution was purchased from Adamas-beta Chemical Co., Switzerland. Deionized water ($18 \text{ M}\Omega \text{ cm}^{-1}$) used in all experiments was obtained by passing through an ultrapure purification system. All reagents were used as received without further purification.

2. *Synthesis of pristine 2H phase MoS_2 .* MoS_2 nanosheets were synthesized using a modified literature procedure.¹ In a typical synthesis, 0.2 mM hexaammonium heptamolybdate tetrahydrate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$) and 6 mM thiourea were dissolved in 10 ml deionized water. Then, the solution was transferred into a 20 ml Teflon-lined stainless steel autoclave and maintained at 200 °C for 24 hours before it was cooled to room temperature. The final black product was washed with ethanol and water for several times and finally dissolved in ethanol for further usage.

3. *Synthesis of pristine 1T phase MoS_2 .* 1T MoS_2 nanosheets were synthesized using a modified literature procedure.² In a typical synthesis, 1 g MoS_2 powder was dissolved in 10 ml 1.5 M n-Butyllithium hexane solution in an Ar filled glove box. Then the solution was sealed in a 20 ml centrifuge tube and sonicated for 3 h. The final black product was washed with hexane and water for several times and finally dissolved in ethanol for further usage.

Supporting Information Figures

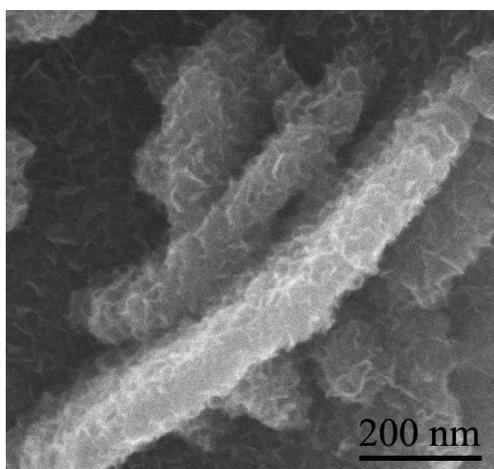


Figure S1. SEM image of edges of MCSO

From the edges of MCSO, it can be inferred that the inner of MCSO is also porous and the feature can give this material a large active area.

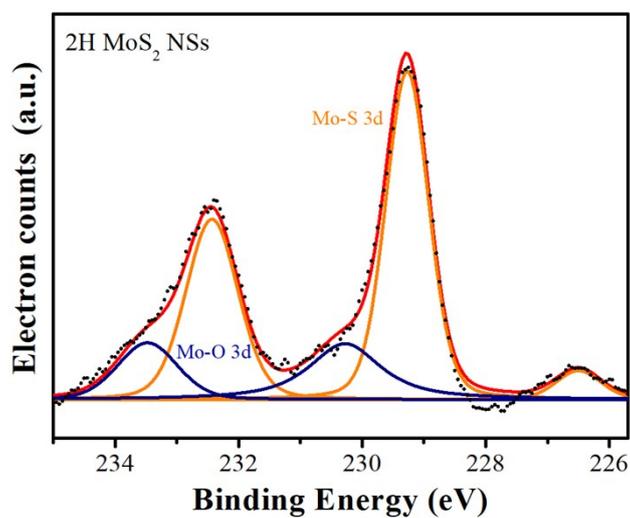


Figure S2. XPS spectra of pristine 2H phase MoS₂.

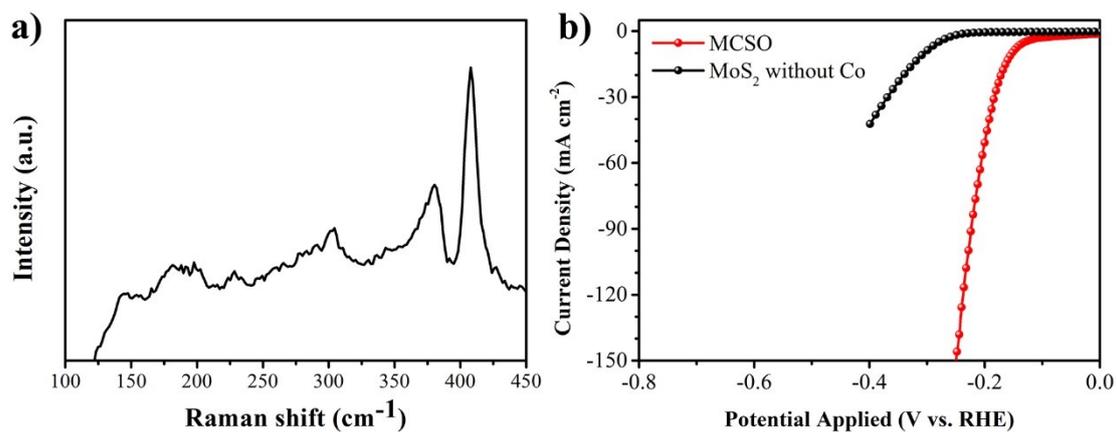


Figure S3. (a) Raman spectrum of MoS₂ synthesized without Co precursors. (b) LSV curves of MCSO and pristine MoS₂.

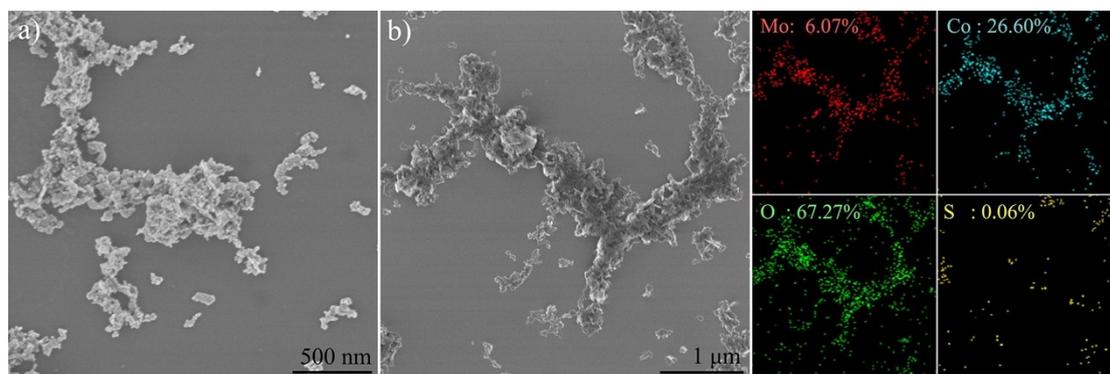


Figure S4. (a) SEM image of MCSO intermediate. (b) EDX mapping image of MCSO intermediate. The numbers shows the content (at%) of different elements.

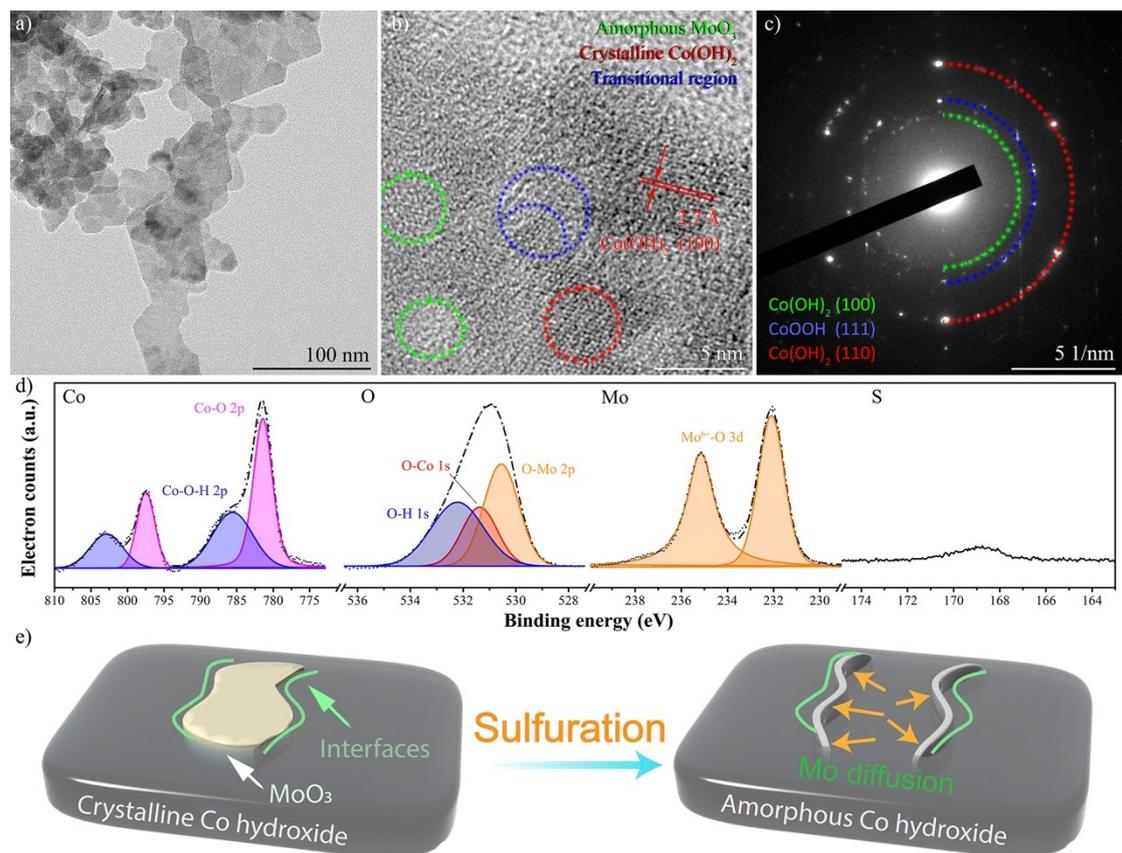


Figure S5. (a) TEM image of MCSO intermediate. (b) HRTEM image of the MCSO intermediate. (c) SAED pattern of the MCSO intermediate. (d) XPS spectra of the MCSO intermediate. (e) Illustration of the formation mechanism of MCSO.

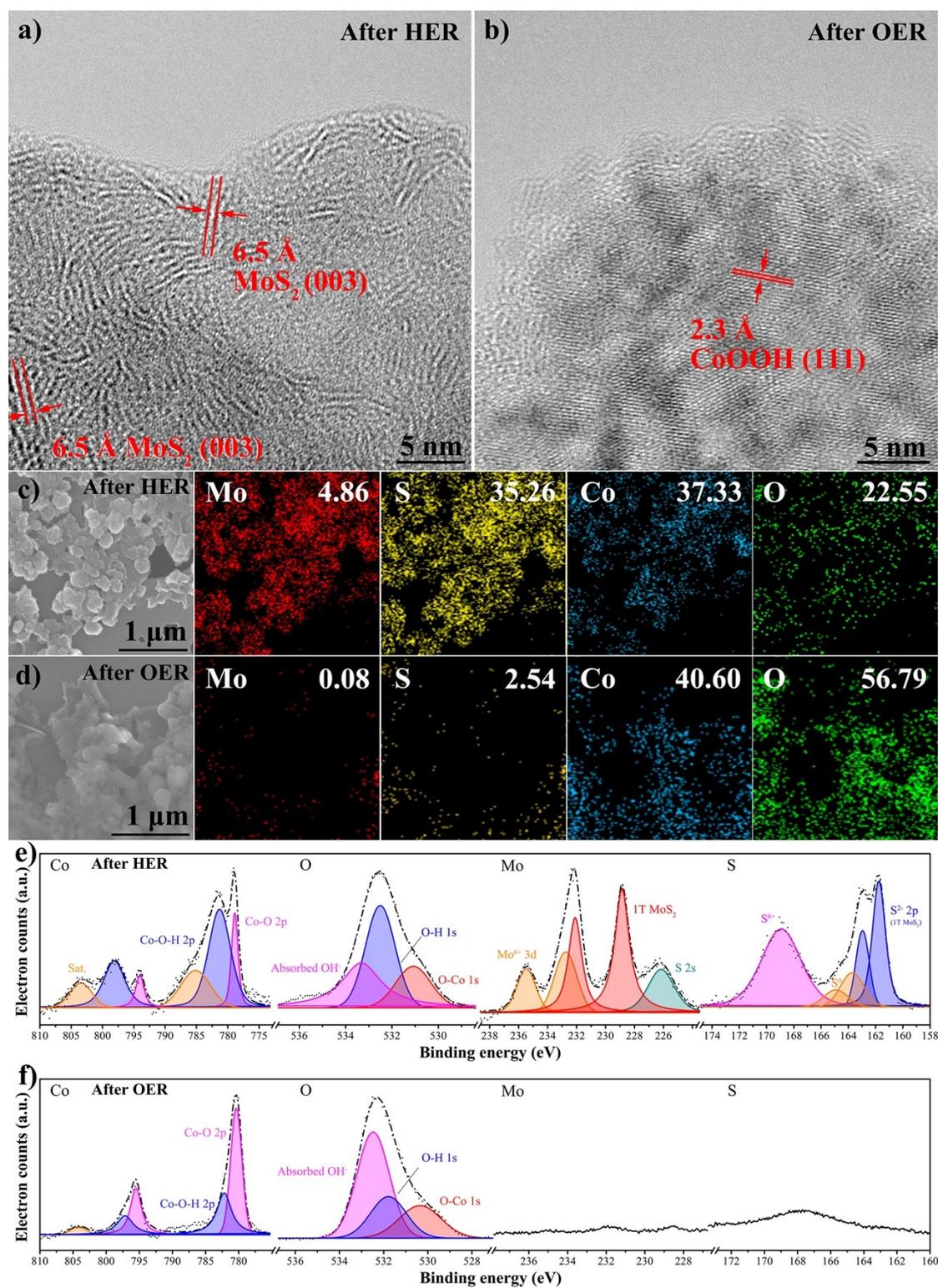


Figure S6. HRTEM image of MCSO after HER (a) and OER (b) stability tests for 25 hours in 0.5 M H_2SO_4 with an applied voltage of -0.16 V and for 20 hours in 1M KOH with an applied voltage of 1.6 V, respectively. (c-d) SEM and EDX mapping image of the corresponding area. The numbers show the atom ratio of different elements. (e-f) XPS spectra of MCSO after HER and OER stability tests.

References

1. J. Xie, H. Zhang, S. Li, R. Wang, X. Sun, M. Zhou, J. Zhou, X. W. D. Lou, Y. Xie, *Adv. Mater.*, 2013, **25**, 5807.
2. D. Voiry, M. Salehi, R. Silva, T. Fujita, M. Chen, T. Asefa, V. B Shenoy, G. Eda, M. Chhowalla, *Nano Lett.*, 2013, **12**, 6222.