

Electronic Supplementary Information

A strategy to synergistically increase the number of active edge sites and the conductivity of MoS₂ nanosheets for hydrogen evolution †

Hailong Yu,^a Xianbo Yu,^a Yujin Chen,^{*a} Shen Zhang,^a Peng Gao,^{*b} and Chunyan Li^{*a}

^aKey Laboratory of In-Fiber Integrated Optics, Ministry of Education, College of Science, Harbin Engineering University, Harbin 150001, China, E-mail: chen yujin@hrbeu.edu.cn and chunyanli@hrbeu.edu.cn

^bCollege of Materials Science and Chemical Engineering, Harbin Engineering University, Harbin 150001, China, E-mail: gaopeng@hrbeu.edu.cn

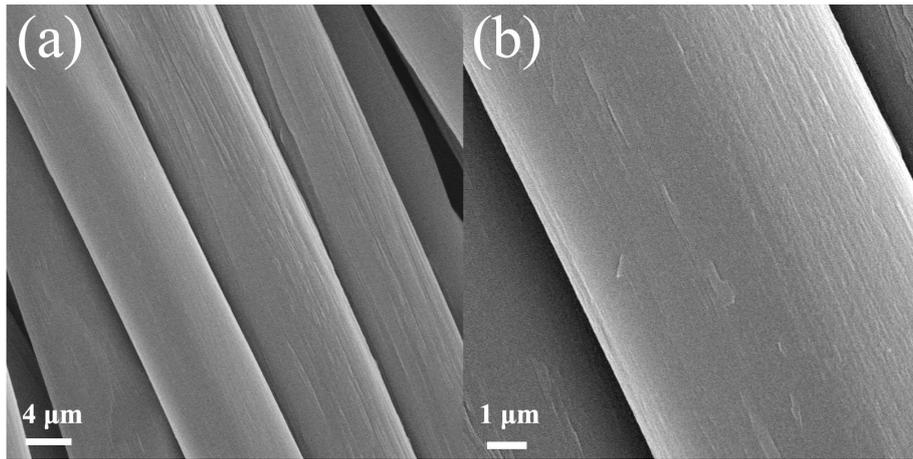


Figure S1 Typical SEM images of CFC at different magnifications, respectively.

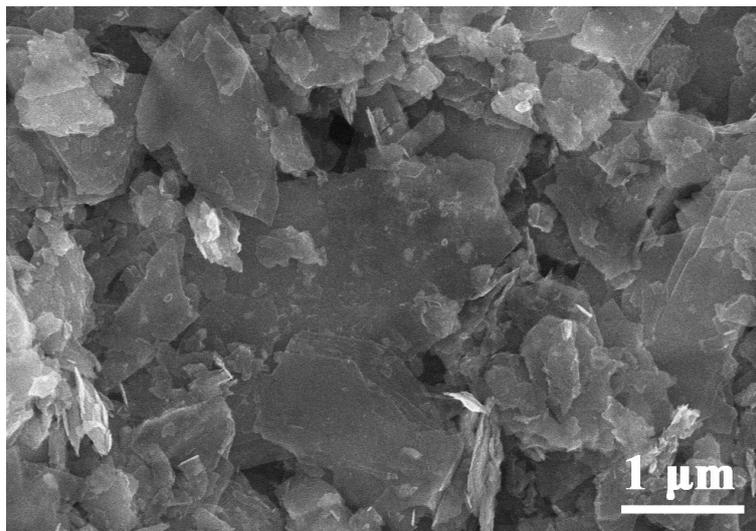


Figure S2 SEM image of bulk MoS₂.

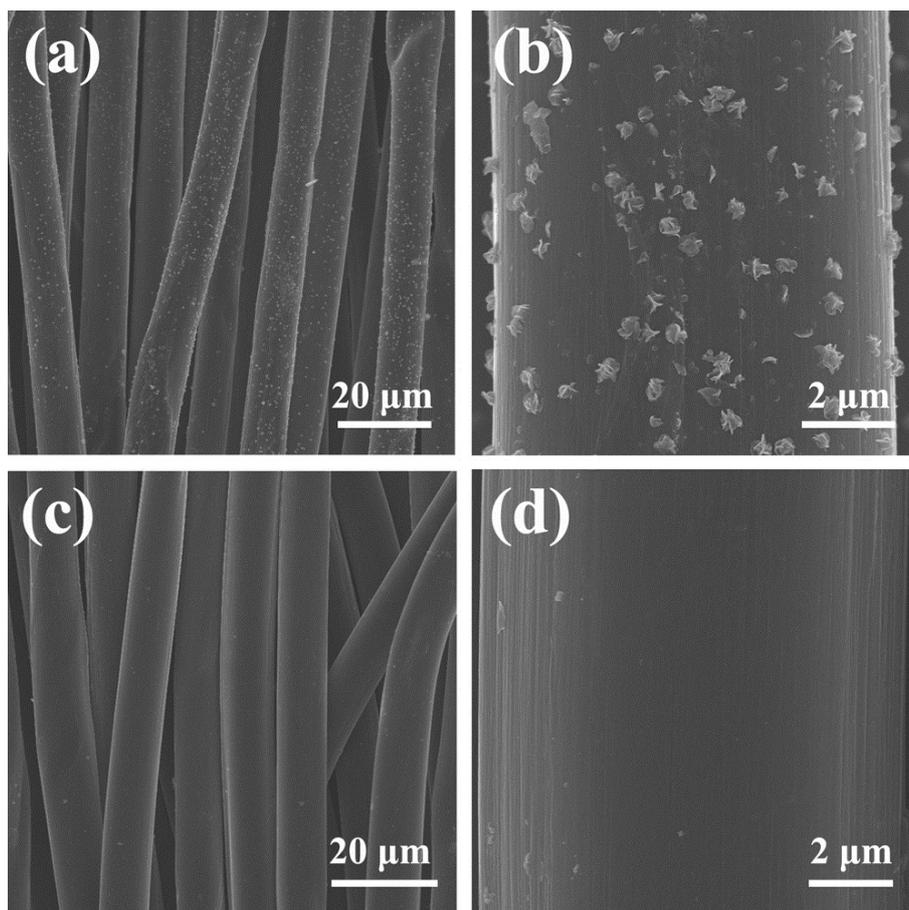


Figure S3 SEM images of samples obtained as MoO_3 replaced with bulk MoS_2 in the presence of NH_4F (a-b) or urea (c-d).

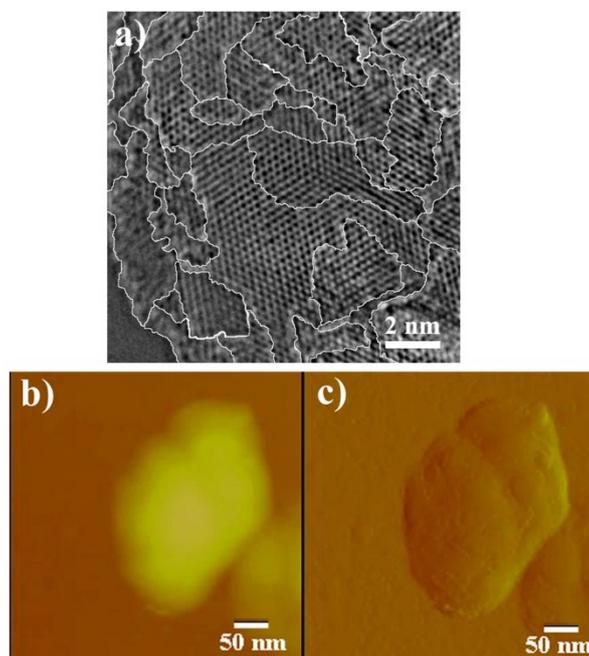


Figure S4 a) HRTEM image with marked pits, b) AFM height, and c) phase images

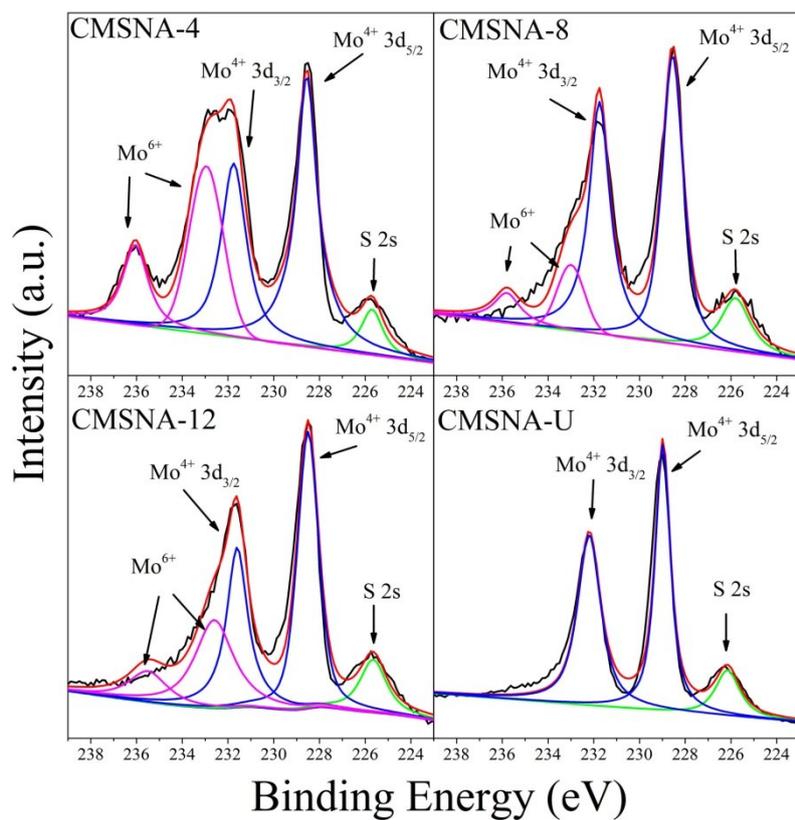


Figure S5 Mo 3d XPS spectra of CMSNs.

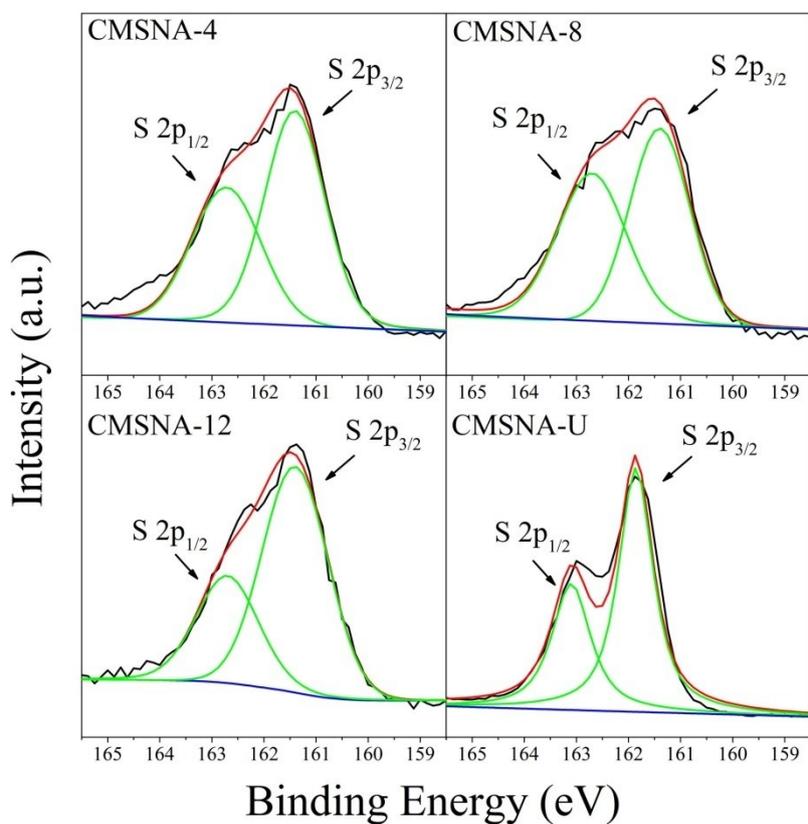


Figure S6 S 2p XPS spectra of CMSNs.

The calculation method for the number of active sites

The absolute components of voltammetric charges (cathodic and anodic) can be obtained from a cyclic voltammetry measurement between -0.2 and 0.6 V (vs. RHE) with 50 mV s⁻¹ scan rate in phosphate buffer solution (PH=7.0). The absolute components of the voltammetric charges reported during one single blank measurement were added. Assuming one electron redox process, this absolute charge was divided by 2. The number of active sites (n) can be calculated with the following equation:

$$n = Q/2F$$

n : Number of active sites (mol).

F : Faraday constant (C mol⁻¹)

Q : The number of voltammetric charges

Table S1 the number of active sites of the CMSNAs

Materials	Number of active sites (10 ⁻⁷ mol cm ⁻²)
CMSNA-4	1.7
CMSNA-8	2.6
CMSNA-12	3.0
CMSNA-U	1.1

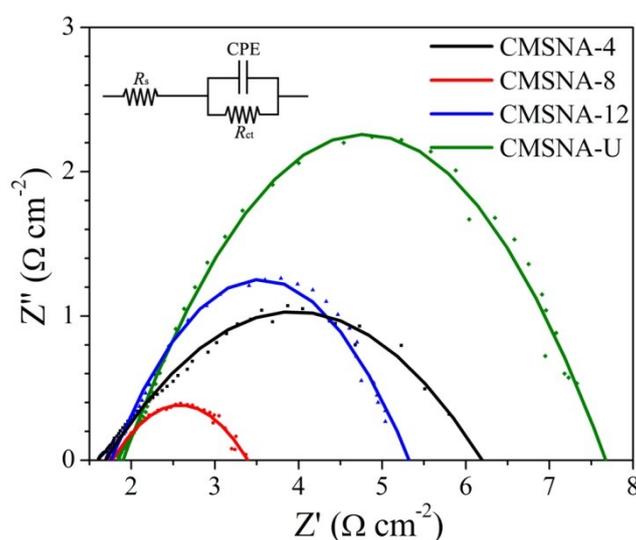


Figure S7 Nyquist plots of impedance spectroscopy analysis of the electrodes, where CMSNA-4, CMSNA-8, CMSNA-12, and CMSNA-U were used as working electrodes. The inset shows a corresponding equivalent circuit.

The synthesis of CMS-H

20 mg of $(\text{NH}_4)_2\text{MoS}_4$ was dispersed into 30 ml N,N-dimethylformamide (DMF). After that carbon fiber cloth was immersed into above solution, the solution was sonicated at room temperature for approximately 10 mins until homogeneous solution was achieved. Then the mixture was transferred into a Teflon-lined stainless steel autoclave with a capacity of 40 mL for hydrothermal treatment at a 200°C temperature for 10 h. The autoclave was cooled down to room temperature naturally, and then the products were washed in distilled water and absolute ethanol under ultrasonication for 10 min, respectively, and dried in a vacuum oven at 60°C.

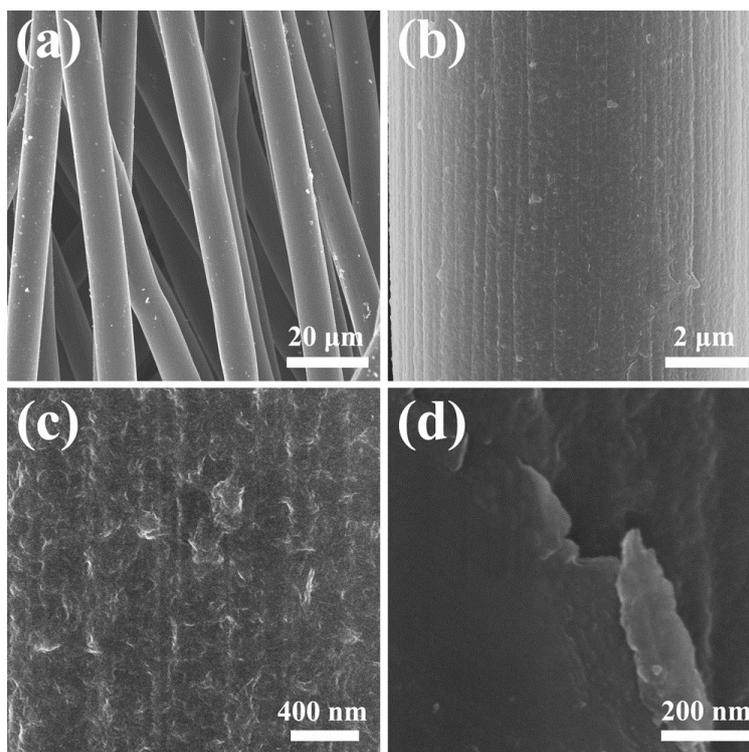


Figure S8 SEM images of CMS-H.

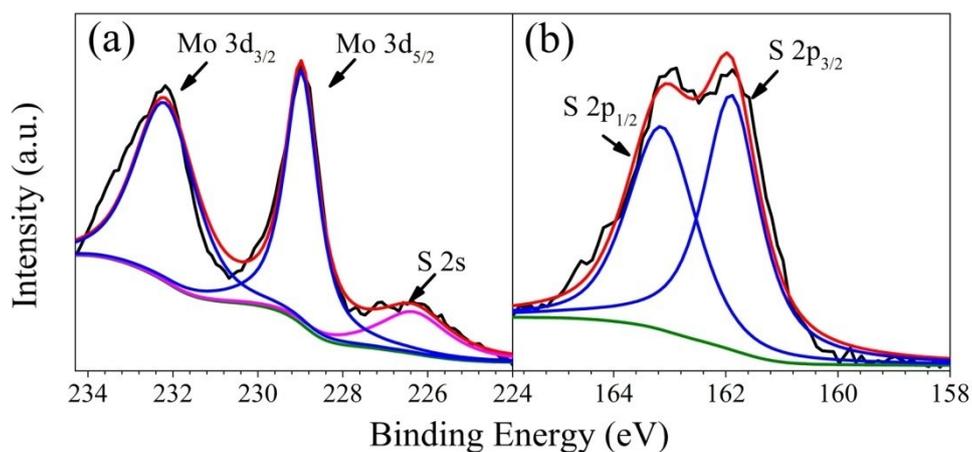


Figure S9 XPS spectrum of CMS-H. (a) Mo 3d and (b) S2p.

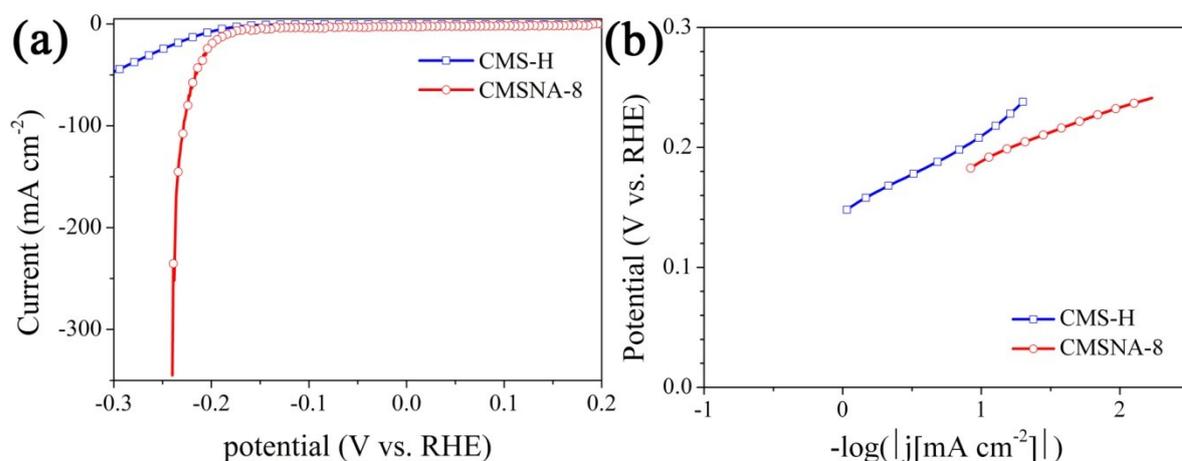


Figure S10 Comparison of HER performance of CMS-H and CMSNA-8. (a) polarization curves and (b) Tafel slopes.

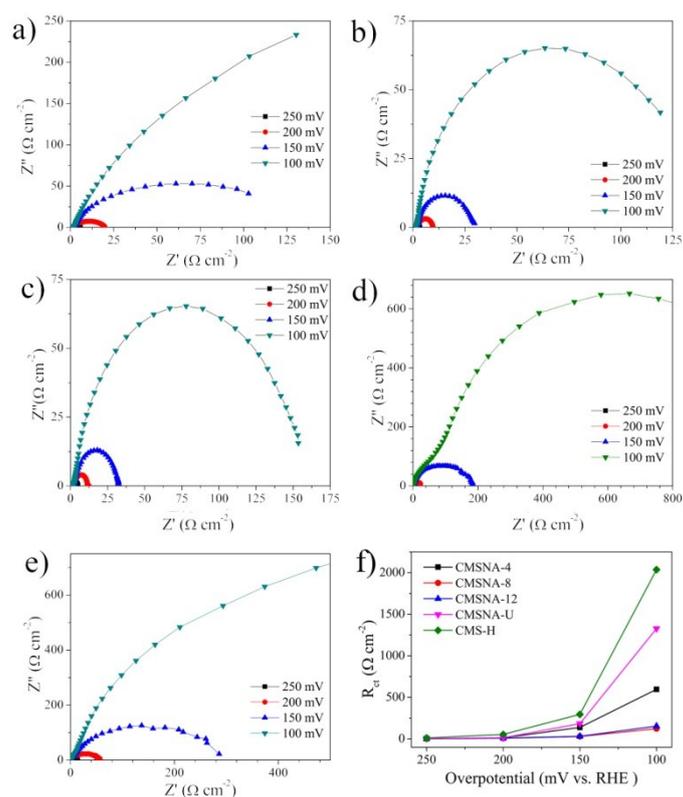


Figure S11 Nyquist plots of impedance spectroscopy analysis of the different samples at various voltages. a) CMSNA-4, b) CMSNA-8, c) CMSNA-12, d) CMSNA-U, e) CMS-H, and f) comparison of R_{ct} values of different samples.

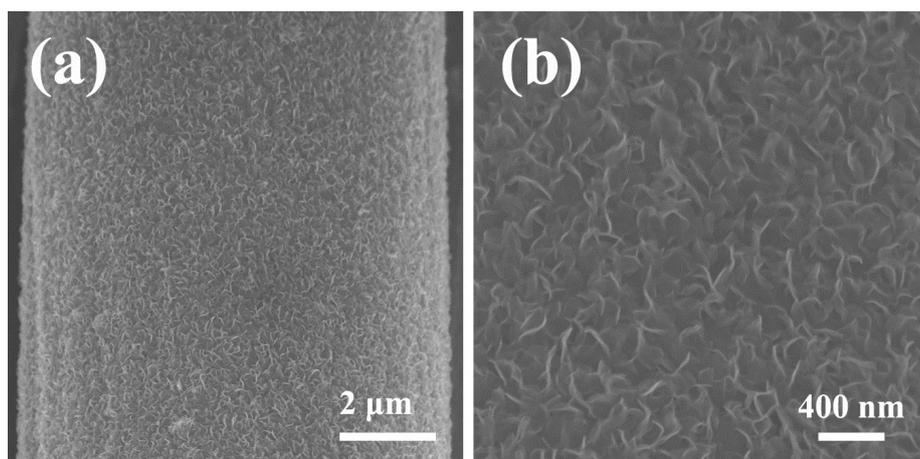


Figure S12 The SEM images of CMSNA-8 after a long-term stability test at an overpotential of 200 mV.

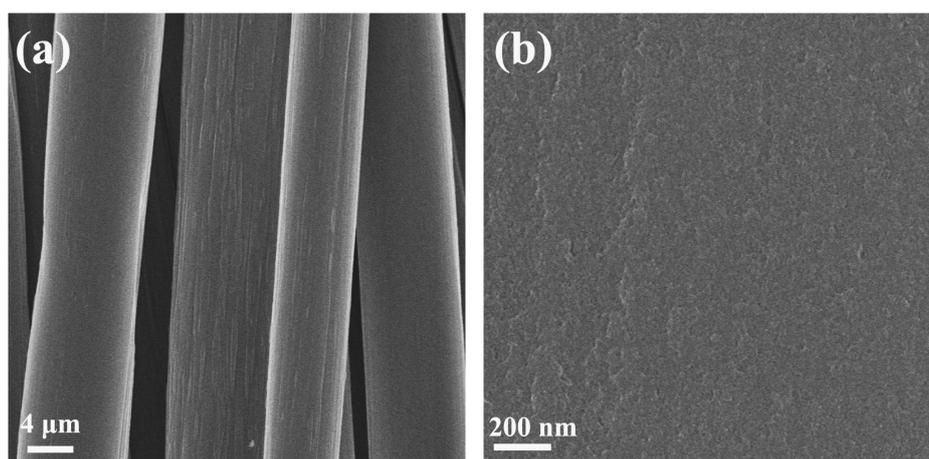


Figure S13 SEM image of CMSNA-8 treated in the HNO₃ solution at 150°C.

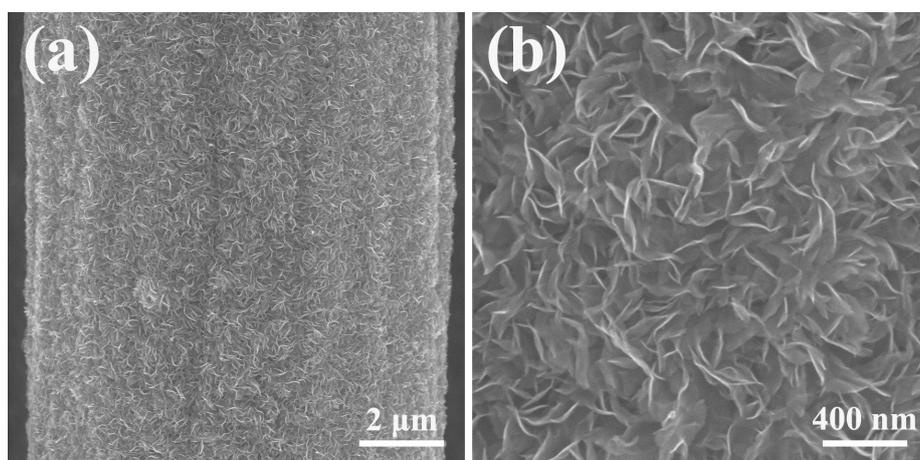


Figure S14 SEM image of MoS₂ nanosheet arrays regrown on CFC under the same conditions as that of CMSNA-8.