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Electronic Supporting Information

for

Hybrid of CuS Nanocrystals Deposited Layered MXene for Efficient Hydrogen Generation

Yi Xie[a*], Md Mushfiqure Rahman[a], Shefiu Kareem[a], Hao Dong[a], Fen Qiao[b*], Wei Xiong[a], Xiaoqing Liu[c], Neng Li[a*], Xiujian Zhao[a]

[a] *State Key Laboratory of Silicate Materials for Architectures, Wuhan University of Technology (WUT), No. 122, Luoshi Road, Wuhan 430070, P. R. China*

[b] *School of Energy & Power Engineering, Jiangsu University, Zhenjiang, 212013, P. R. China*

[c] *Center of Materials Research & Testing, Wuhan University of Technology, Wuhan, Hubei 430070, P.R. China.*

Email: xiey@whut.edu.cn (Yi Xie); fqiao@ujs.edu.cn (Fen Qiao); lineng@whut.edu.cn (Neng Li).

Synthesis of Multilayered MXene

Multilayered MXene ($Ti_3C_2T_x$) colloidal suspension was prepared using a modified procedure of etching Ti_3AlC_2 powder in HF solution (49%) previously reported by Sun et al.[1] Typically, 3 g of Ti_3AlC_2 powder was slowly added into a Teflon lined bottle containing 60 ml HF solution at room temperature (RT) under magnetic stirring, followed by sealing the Teflon-lined bottle and increasing the temperature to 30°C or 60°C with a ramp around 5°C/min in a water bath. The reaction was performed under stirring for 24 h, and the resulting black dispersion was transferred into plastic tubes, which was then washed repeatedly by adding distilled H_2O until the pH reached 6-7 and centrifuged at 3500 rpm for 5 minutes. The supernatant was disposed and the final precipitate was washed with ethanol and dried in a vacuum oven at 60 °C for 24 h to achieve multilayered MXene.

Synthesis of Single-layered MXene

Single-layered MXene was produced by intercalating multilayered $Ti_3C_2T_x$ with TMAOH (25% in H_2O).[2] Typically, 0.2 g of the as-synthesized multilayered MXene was dispersed in 20 mL TMAOH under stirring at RT for 24 h, followed by swaying with hands. The resulting clay-like sediment (i.e. single-layered or few-layered MXene) was collected by washing and centrifuging at 3500 rpm for 15 min.

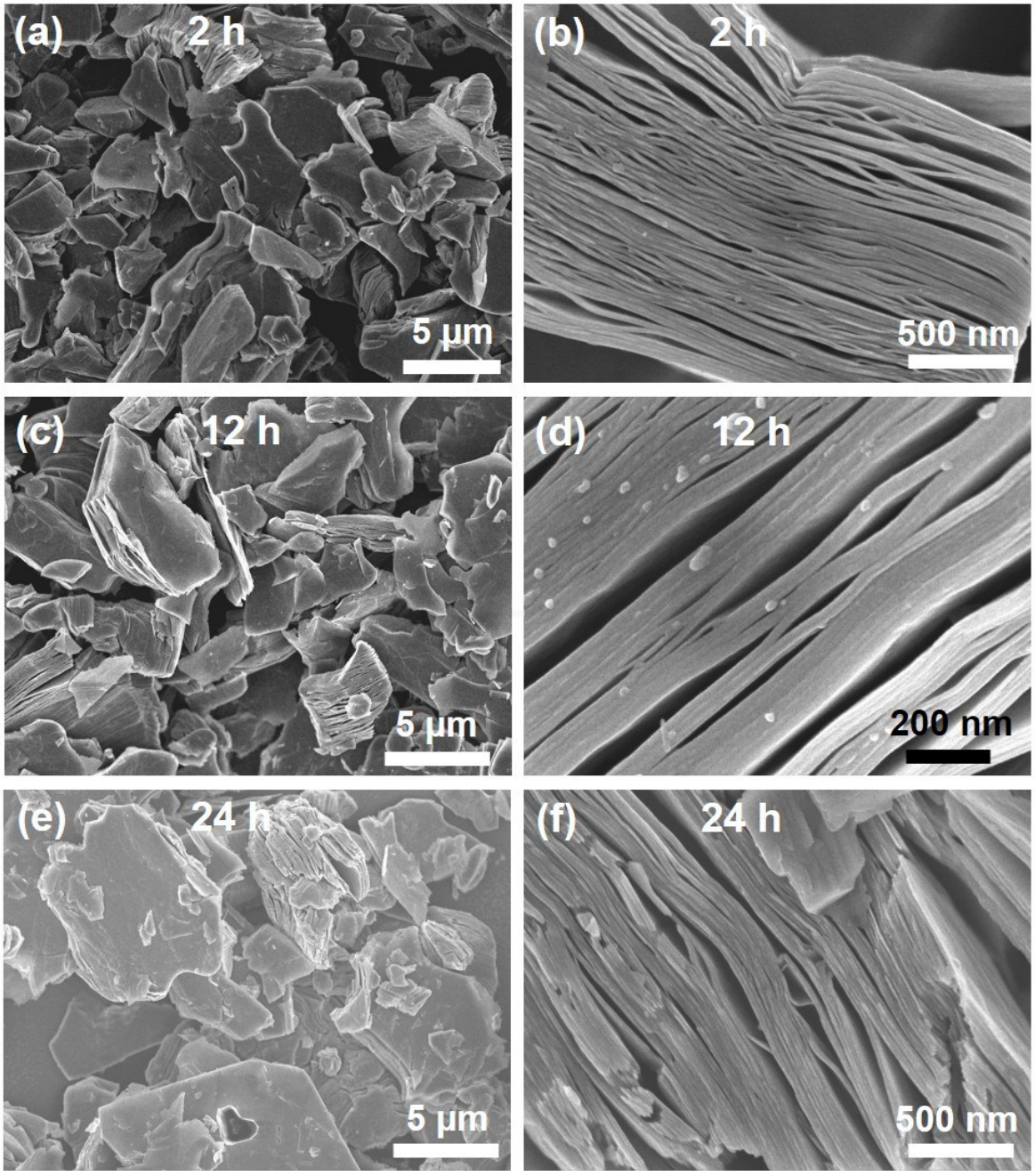

Fig. S1 *FE-SEM and magnified FE-SEM images of multi-layered MXene collected at 30°C for different reaction time as dictated.*

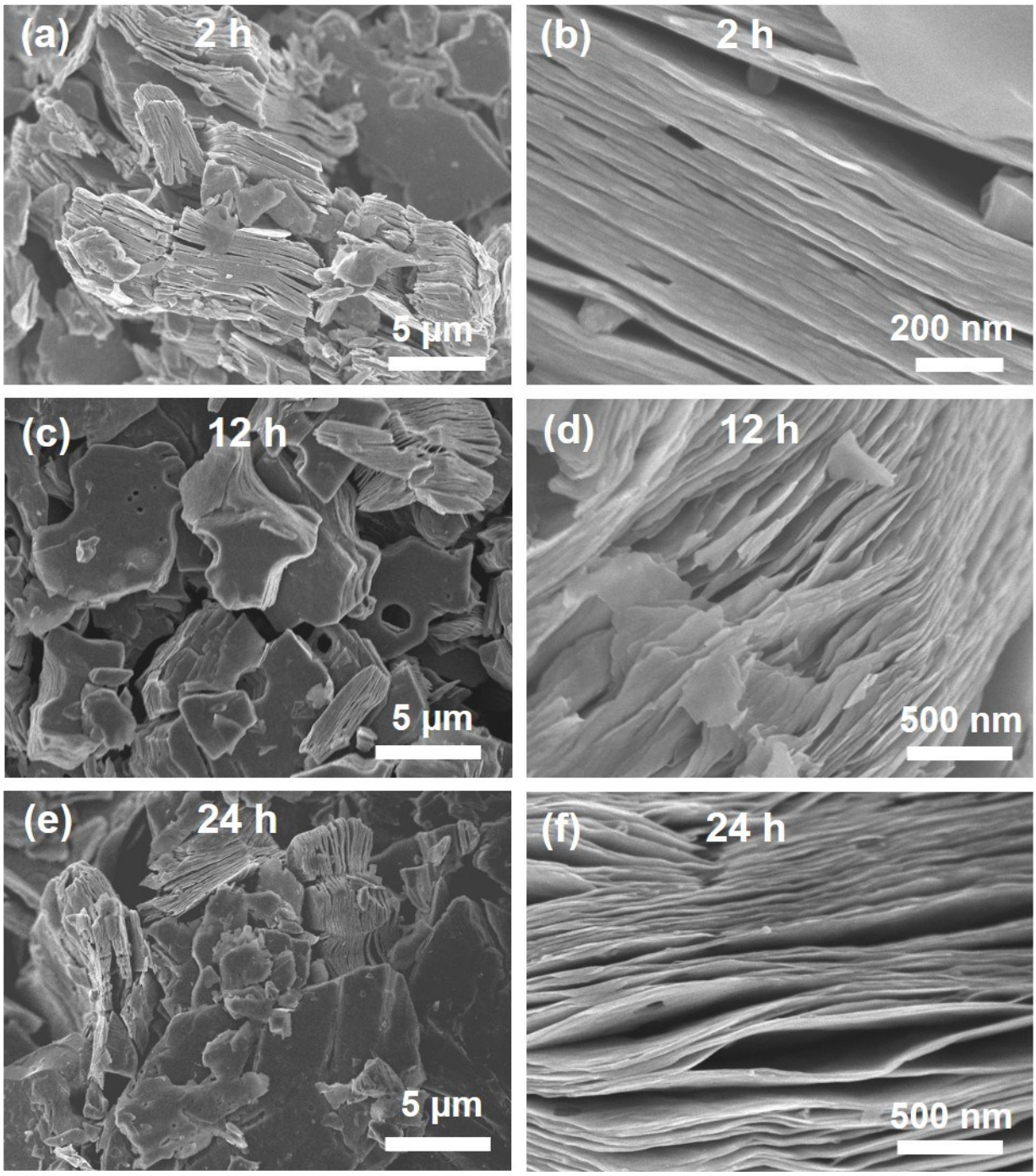

Fig. S2 *FE-SEM and magnified FE-SEM images of multi-layered MXene collected at 60°C for different reaction time as dictated.*

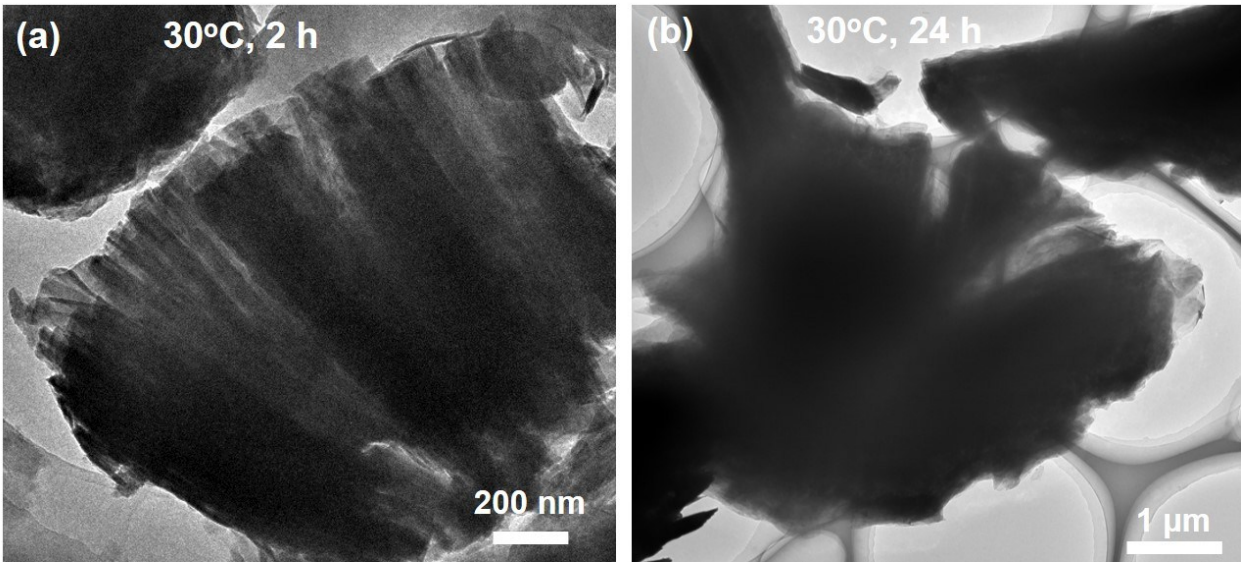

Fig. S3 *TEM images of multi-layered MXene collected at 30°C for different reaction time as dictated.*

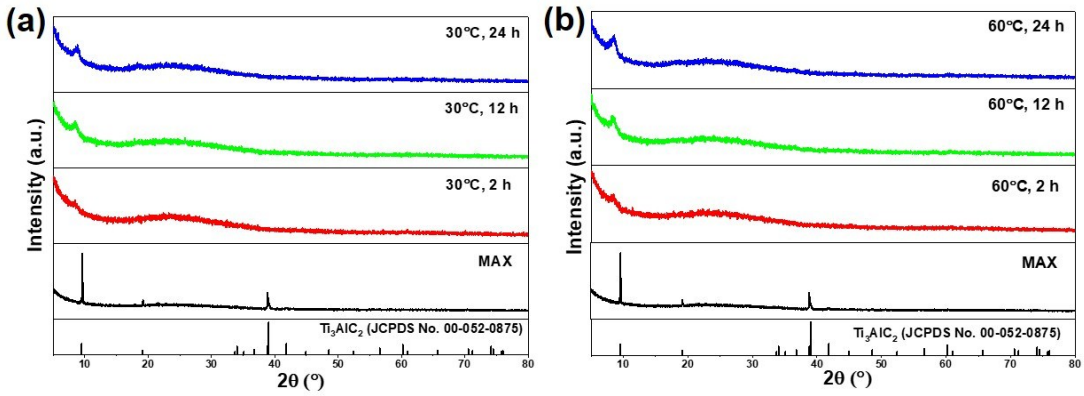

Fig. S4 *XRD patterns of multi-layered MXene collected at 30°C (a) and 60°C (b) for different reaction time as dictated.*

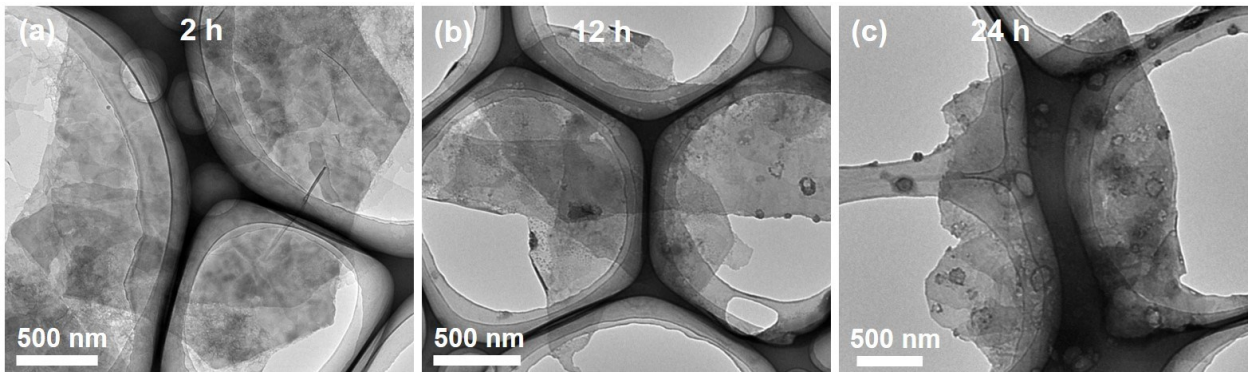

Fig. S5 *TEM images of single-layered MXene collected at room temperature for different reaction time as dictated.*

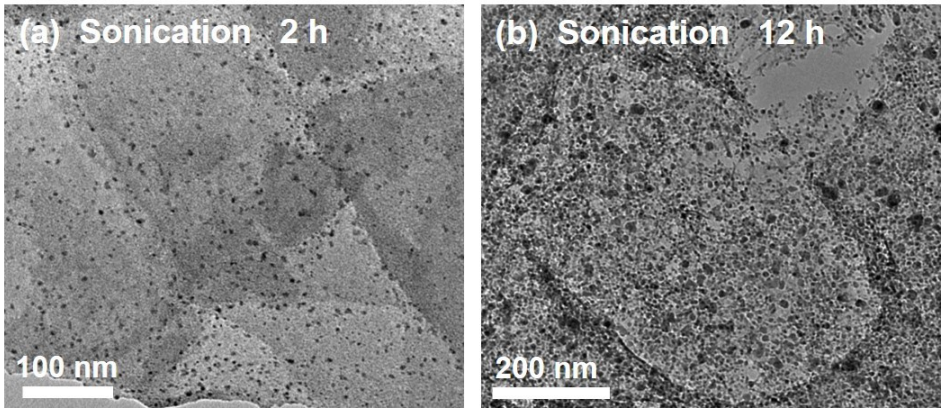

Fig. S6 *TEM images of MXene collected by sonicating the TMAOH-intercalated layered MXene for different time as dictated.*

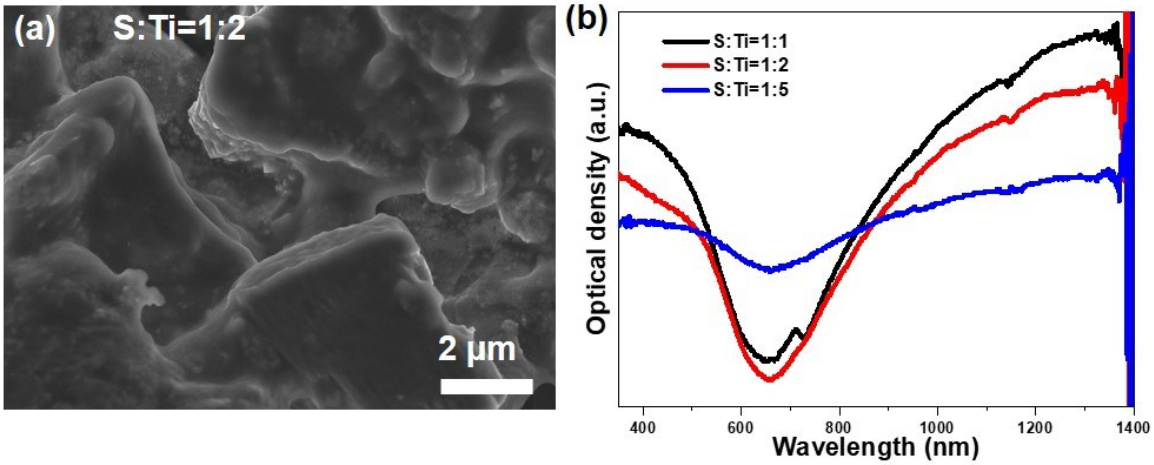

Fig. S7 SEM image (a) and absorption spectra (b) of CuS NCs-anchored few-layered MXene after ligands exchange and converting to water phase.

References

1. Sun, D.; Wang, M.; Li, Z.; Fan, G.; Fan, L.-Z.; Zhou, A., Two-dimensional Ti_3C_2 as anode material for Li-ion batteries. *Electrochem. Commun.* **2014,** *47*, 80-83.

2. Wang, Z.; Xuan, J.; Zhao, Z.; Li, Q.; Geng, F., Versatile Cutting Method for Producing Fluorescent Ultrasmall MXene Sheets. *ACS Nano* **2017,** *11* (11), 11559-11565.