Electronic Supplementary Information

Controlled Synthesis of Mo-doped Ni$_3$S$_2$ Nano-rod: an Efficient and Stable Electro-catalyst for Water Splitting

Zheng Cui$^1$, Yuancai Ge$^1$, Hang Chu$^1$, Robert Baines$^2$, Pei Dong$^2$, Jianhua Tang$^1$, Yang Yang$^1$, Pulickel M. Ajayan$^2$, Mingxin Ye$^1$$^*$, Jianfeng Shen$^1$$^*$

$^1$ Institute of Special Materials and Technology, Fudan University, Shanghai, 200433, P. R. China

$^2$ Department of Materials Science and NanoEngineering, Rice University, 6100 Main Street, Houston, TX 77005, USA

**Figure S1.** Digital images of electrode surface of 200-SMN/NF (a) before and (b) after hydrothermal process.

Figure S1 shows digital images of the Ni foam surface before and after Mo-doped Ni$_3$S$_2$ growth through the one-step hydrothermal method. The original color of the Ni foam electrode is brown (Figure S1a); once the Mo-doped Ni$_3$S$_2$ nano-rod arrays grow on the Ni foam electrode, the foam surface becomes completely black (Figure S1b). This change in color indicates the complete and uniform formation of Mo-doped Ni$_3$S$_2$ on the Ni foam.
Figure S2. XRD patterns of Ni$_3$S$_2$/NF

Figure S3. Raman spectrum of 180-AMN/NF, 200-AMN/NF, 220-AMN/NF
Figure S4. Raman spectrum of 180-SMN/NF (a), 200-SMN/NF (b), 220-SMN/NF (c), Ni$_3$S$_2$/NF (d)

In Figure S3, besides the Raman resonance bands related to Ni$_3$S$_2$, three Raman resonance bands at 820, 891 and 939 cm$^{-1}$, ascribed to the MoO$_x$ structure are evident. However, in Figure S4, the three Raman resonance bands at 820, 891 and 939 cm$^{-1}$ are not visible. These results, together with the HER and OER results, indicate that Mo-doped Ni$_3$S$_2$ using sodium molybdate as Mo source presents superior HER and OER activities to the Mo-doped Ni$_3$S$_2$ using ammonium molybdate as Mo source, because ammonium molybdate lead to the formation of MoO$_x$.[1]
Figure S5. SEM images and EDX-mapping of Ni$_3$S$_2$/NF

Figure S6. EDX spectrum of Ni$_3$S$_2$/NF
Figure S7. SEM images and EDX-mapping of 180-AMN/NF

Figure S8. EDX spectrum of 180-AMN/NF
**Figure S9.** SEM images and EDX-mapping of 200-AMN/NF.

**Figure S10.** EDX spectrum of 200-AMN/NF
Figure S11. Nyquist plots of, 180-SMN/NF, 200-SMN/NF, 220-SMN/NF, 180-AMN/NF, 220-AMN/NF, Ni$_3$S$_2$/NF, and bare Ni foam electrodes recorded at an applied potential of -1.2 V with a frequency range of 10 kHz to 10 mHz in 1 m KOH.

References: