Electronic Supplementary Information for

Physical Vapor Deposition of Amorphous MoS_2 Nanosheet Arrays on Carbon Cloth towards Highly Reproducible Large-Area Electrocatalysts for Hydrogen Evolution Reaction

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Experimental Section

Synthesis of a-MoS_2 NA/CC

Carbon cloth (CC, Wuhan Instrument, Wuhan) was carefully cleaned with concentrated HNO_3 to remove impurities on surface, then rinsed by deionized water and ethanol before use. The deposition of a-MoS_2 was carried out on a magnetron sputtering system (PVD 75, Kurt J. Lesker, USA) using molybdenum sulfide (99.99%, Purui, Beijing) as the target. The deposition time was kept at 1000 s. The sputtering power was adjusted from 70 W to 150 W to control the morphology of the deposited a-MoS_2. In order to keep the same loadings of MoS_2 for all the samples deposited at different power, we first measured the thickness of the MoS_2 thin film deposited on Si substrate in parallel with the samples to calculate the sputtering rate at different power. Then, we calibrated the sputtering time to obtain MoS_2 nanostructures on carbon cloth with the same loading of MoS_2 on carbon cloth by weighing the sample before and after the sputtering process.

Electrochemical measurements

All electrochemical measurements were performed on a 760E bipotentiostat (CHI instruments) in a typical three electrode setup. In a typical measurement, a-MoS_2 NA/CC (0.5 × 0.5 cm) with a MoS_2 loading of ~0.41 mg cm^{-2}

was used as a working electrode, a graphite rod as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode. The electrolyte solution is 0.5 M H_2SO_4. Another two a-MoS_2 nanostructured thin films obtained at different sputtering power with same loading (~0.41 mg cm^{-2}) and commercial Pt/C catalyst (20 wt.% Pt, Johnson Matthey, 0.35 mg cm^{-2}.) were also measured for comparison. In all measurements, the SCE reference electrode was calibrated with respect to reversible hydrogen electrode (RHE). LSV measurements were conducted at a scan rate of 2 mV/s. All the potentials reported in our work were versus RHE.

Characterizations

Morphology analysis was conducted using a Hitachi S-4800 field emission scanning electron microscope operated at 10 kV. Elemental mapping and electron energy dispersive spectroscopy (EDS) were performed with a EDS detector equipped on the Hitachi S-4800 SEM. Transmission electron microscopy (TEM) and high resolution TEM (HRTEM) images were recorded on a JEOL-2100F microscope operating at 200 kV. X-ray diffraction (XRD) experiments was carried out on a PANalytical high resolution X-ray diffraction system (model EMPYREAN) with a Cu Kα radiation (λ = 0.15406 nm). X-ray photoelectron spectroscopy (XPS) was recorded on a Thermo Scientific ESCALab250Xi using 200W monochromated Al Kα radiation.

Figure S1

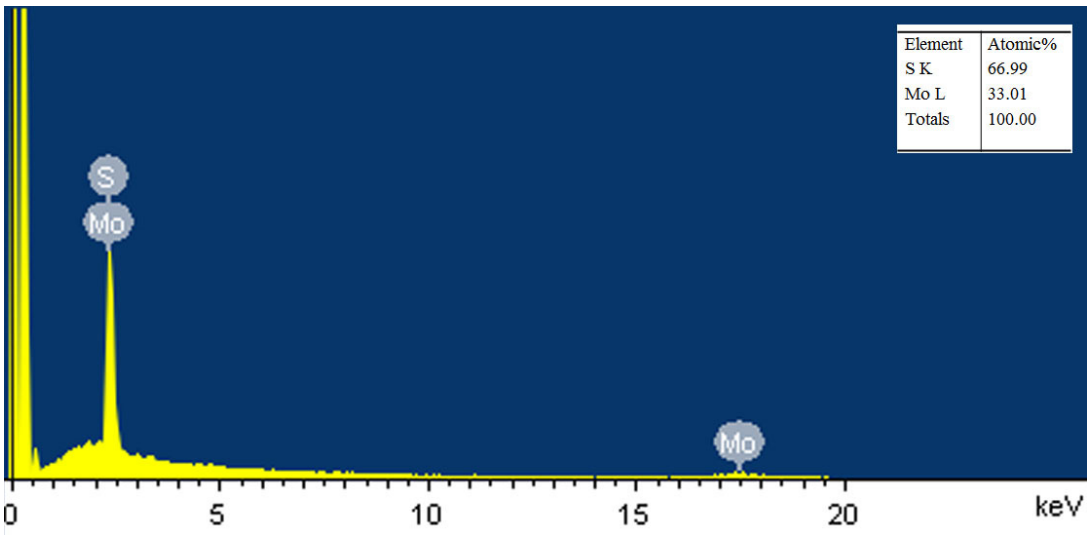

Fig. S1. A typical EDS spectrum of a-MoS_2 NA/CC.

Figure S2

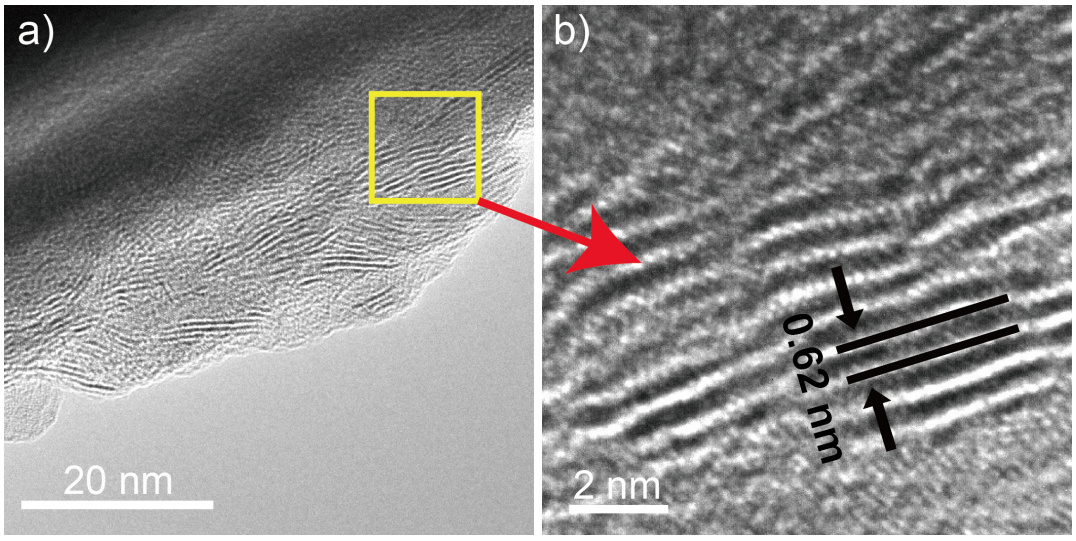

Fig. S2. a) Low, b) high magnification TEM images of a-MoS_2 NA/CC.

Figure S3

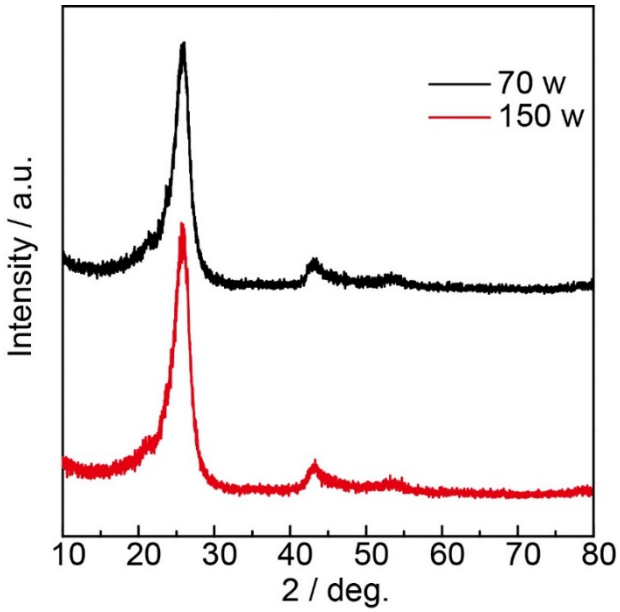

Fig. S3. XRD patterns of the as-prepared a-MoS_2 nanostructured thin films at different sputtering powers.

Figure S4

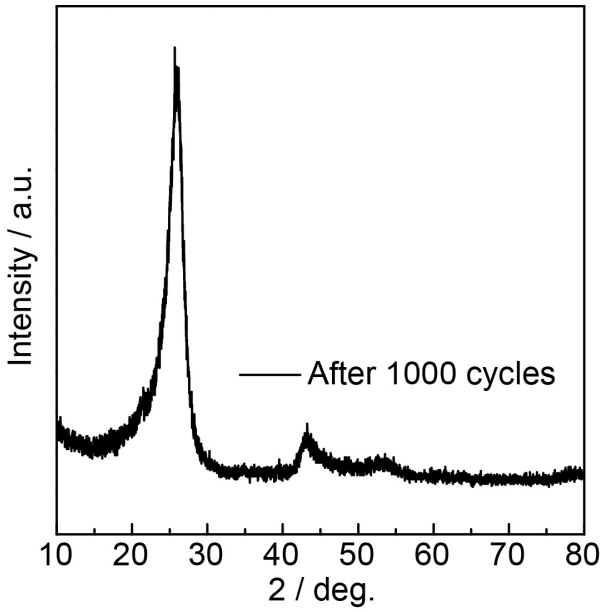

Fig. S4. XRD pattern of a-MoS_2 NA/CC after aging test of 1000 CV scans.

Figure S5

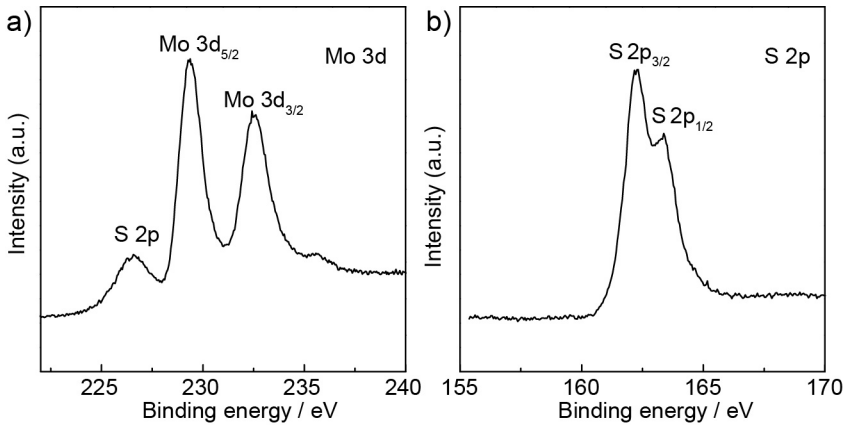

Fig. S5. XPS spectra of a-MoS_2 NA/CC after 1000 CV scans: a) high resolution Mo 3d spectrum; b) high resolution S 2p spectrum.