Electronic Supplementary Material (ESI) for Sustainable Energy & Fuels.

Supporting information

Few-layered ultrasmall MoS_2 nanosheets cathode material for high performance rechargeable aluminum-ion battery

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Experiment Section

Preparation of the samples

The M-MoS_2 was prepared through microwave-assisted precursor synthesis method combined with subsequent calcination. Typically, 30 mL of PEG-400 was added into a 300 mL three-necked flask, and then 0.5004 g Sodium molybdate dehydrate and 0.6200 g Thiourea were added with vigorous stirring for 10 min. Afterwards, the mixed solution were transferred to a multipurpose microwave chemical synthesizer X-8000, following with microwave irradiation 800 W for 6 min under 185°C with vigorous stirring. After natural cooling, the product was washed thoroughly with ethanol and water several times via centrifugation. Then, the resultant product was subjected to drying at 60 °C for 6 h. Finally, the M-MoS_2 was obtained after calcination at 800 °C for 2 h under argon atmosphere. For comparison, the H-MoS_2 and S-MoS_2 samples were also synthesized by hydrothermal method and solvothermal method at 185 °C for 20 h using deionized water and PEG-400 as solvent, respectively.

Characterization

The crystal characteristics of the M-MoS_2, H-MoS_2 and S-MoS_2 samples were characterized using X-ray diffraction (XRD) on an X-ray 6000 diffractometer with the 2θ angle region from 10° to 70° at a scan rate of $6°\ min^{-1}$. The morphological features of the samples were determined by scanning electronic microscopy (SEM), transmission electron microscopy (TEM) and high-resolution TEM (HR-TEM) which were carried out using JSM-7500F (5 kV) and

JEM-2100F (JEOL, 200 kV) instruments. N_2 adsorption–desorption isotherms were obtained using a NOVA 2200e system. X-ray photoelectron spectrometer (XPS) analyses were performed using an ESCALAB 250 instrument.

Electrochemical testing

2032-type coin cells were assembled in an argon filled glovebox to estimate the electrochemical performances of the M-MoS_2, H-MoS_2 and S-MoS_2 samples. The active material, acetylene black and poly(vinylidene fluoride) (PVDF) were added into the N-methylpyrrolidone (NMP) solvent dispersant with a weight ratio of 8:1:1 to prepare cathode slurries. The positive electrodes were prepared by coating the slurries onto Ta foil (1.5cm^2) and dried at 60 °C for 12 h in a vacuum. The Al metal (30μm) was used as the counter electrode and the Whatman glass fiber (GF/C) was used as the separator to assemble the cells. A room-temperature ionic liquid electrolyte was made by mixing anhydrous aluminum chloride ($AlCl_3$) and 1-ethyl-3-methylimidazolium chloride ([EMIm]Cl) at a molar ratio of 1.3:1 in a glove box filled with high purity argon gas. A LAND-CT2001A battery test system was used to test the charge–discharge curves in a voltage range of 0.01–2.0 V. The specific capacities at different current densities were calculated based on the mass of active materials (about 1.2 mg cm^{-2}). Cyclic voltammetry (CV) curves were obtained in a voltage range of 0.01–2.0 V at a scan rate of 0.2 mV s^{-1}. The electrochemical impedance spectra (EIS) were measured in the frequency range of 0.1 Hz–1 MHz with a 5 mV amplitude. The CV and EIS measurements were performed on a CHI660D electrochemical workstation.

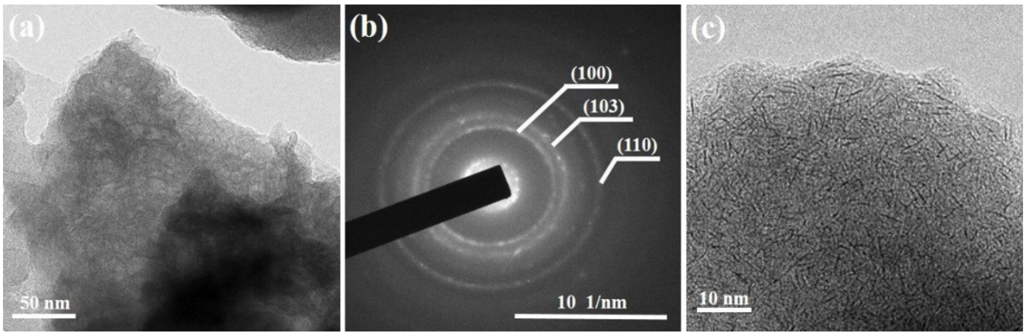

Fig. S1 (a) The TEM, (b) SAED pattern and (c) HR-TEM image of M-MoS_2.

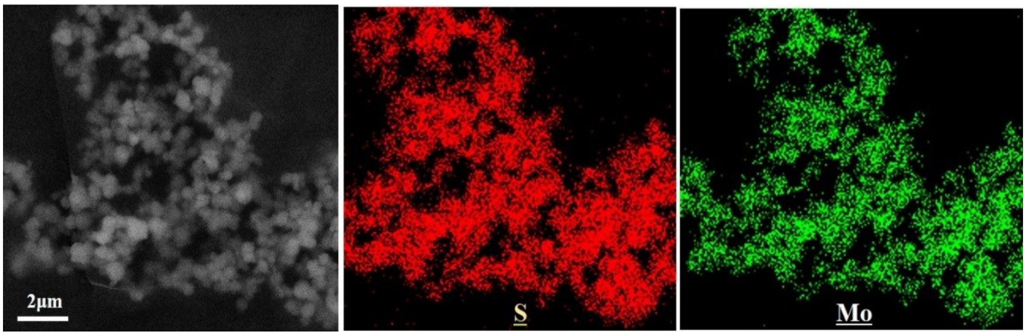

Fig. S2 The SEM image and its related elemental mapping of S and Mo images of the M-MoS_2.

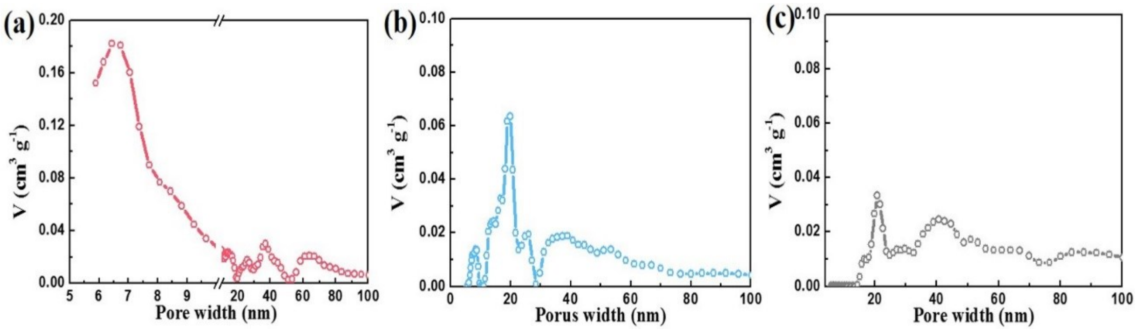

Fig. S3 The pore size distribution of the (a) M-MoS_2, (b) S-MoS_2 and (c) H-MoS_2.

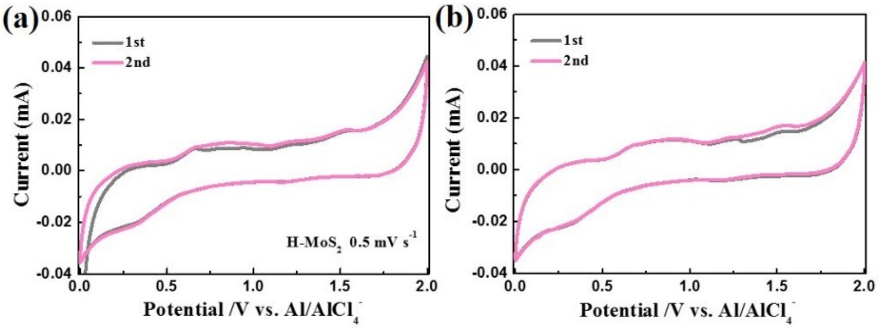

Fig. S4 The CV curves at 0.5 mV s^{-1} for the first two cycles of (a) S-MoS_2 and (b) H-MoS_2.

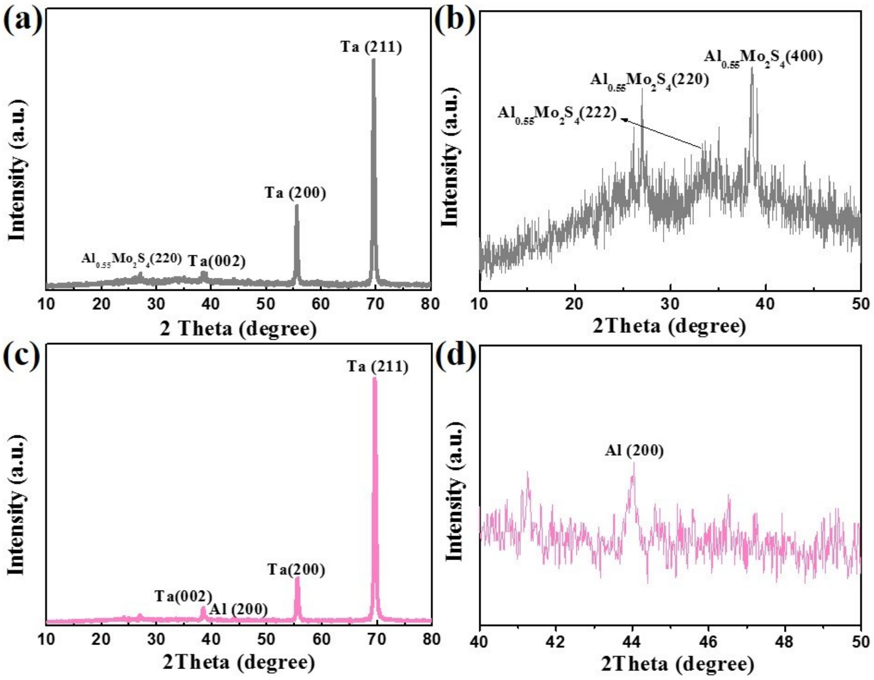

Fig. S5 The XRD pattern of M-MoS_2 after discharge to 0.6V (a, b) and after charge to 1.15V (c, d).

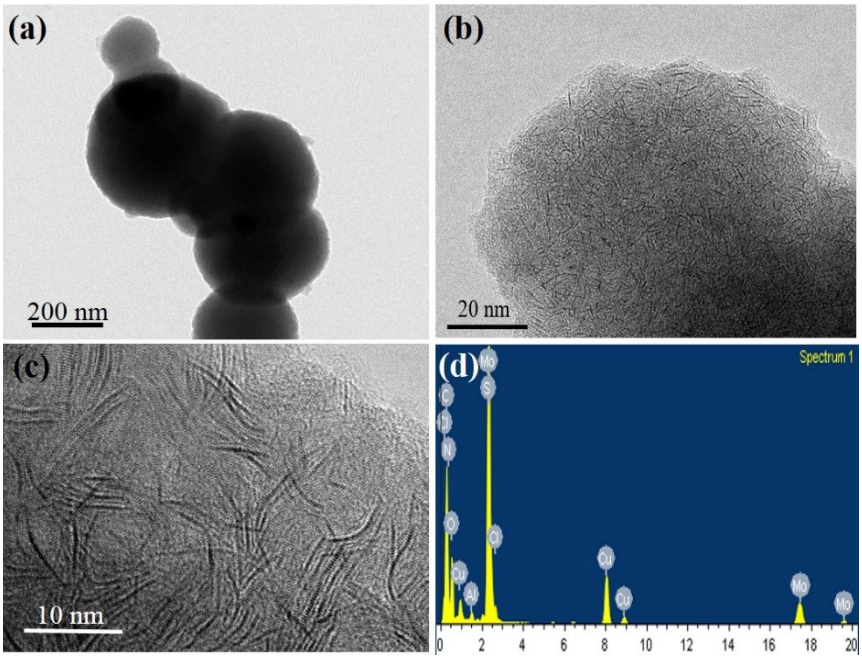

Fig. S6 The TEM (a,b), HR-TEM (c) and EDS (d) images of M-MoS_2 after 100 cycles.

Table 1 The comparison list of Sulfide-based cathode materials for AIBs.

Sample	Specific capacity (mAh g^{-1})	Current density (mA g^{-1})	Reference	Journal
G-SnS_2	**70** after 100 cycles	200	14	Adv. Mater. (2017)
Ni_3S_2@G	**60** after 100 cycles	100	22	Adv. Energy Mater. (2016)
CuS@C	**90** after 100 cycles	20	23	ACS Nano (2017)
Co_3S_4	**100** after 150 cycles	50	24	Nano Energy (2019)
MoS_2@C	**126.6** after 200 cycles	100	25	ACS Sustainable Chem. Eng. (2019)
Bulk MoS_2	**66.7** after 100 cycles	40	26	ACS Appl. Mater. Interfaces (2018)
M-MoS_2	**73** after 150cycles	100	-	Our Work