

**Electronic Supplementary Information for**  
**Template Synthesis of Hollow MoS<sub>2</sub>-Carbon Nanocomposites**  
**Using Microporous Organic Polymers and Their Lithium Storage Properties**

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**Experimental Section**

TEM and HR-TEM images were obtained using a JEOL 2100F unit operated at 200kV. Adsorption-desorption isotherms for N<sub>2</sub> (77 K) were recorded by using BELSORP II-mini volumetric adsorption equipment. Pore size analysis was conducted by DFT method. PXRD patterns were obtained using a Rigaku MAX-2200 and filtered Cu-K $\alpha$  radiation. XPS spectra were obtained using a Thermo VG and Monochromatic Al-K $\alpha$  radiation. Elemental analysis was performed on a CE EA1110 instrument.

**Synthetic procedure for MSC-2**

Hollow organic template (**H-MOP**) was prepared based on the procedure in our previous paper (ref. 11a in the text). The detail synthetic procedure for the **H-MOP** used in this study is as follows. First, as templates for the **H-MOP**, the silica spheres (diameter 120~130 nm) were prepared. In a 500 mL flask, ethanol (200 mL), ammonia solution (4 mL, 28~30 % in water), and water (2.7 mL) were mixed by stirring for 5 minutes at room temperature. Tetraethyl orthosilicate (4.4 mL) was added to the mixture. The reaction mixture was stirred vigorously for 18 hours at room temperature. After ~4 hours, transparent solution became milky white color. After 18 hours, the 1:3 mixture of methylene chloride and hexane (350 mL) was added. The resultant white precipitates, silica spheres were collected by centrifugation, washed with ethanol and water, and dried under vacuum.

In a 100 mL Schlenk flask, the silica spheres (0.60 g) were dispersed in triethylamine (60 mL). Bis(triphenylphosphine)palladium dichloride (17 mg, 0.024 mmol) and copper iodide (8.8 mg, 0.024 mmol) were added. The mixture was treated by sonication for 1 hour at room temperature. Tetrakis(4-ethynylphenyl)methane (0.10 g, 0.24 mmol) and 1,4-diiodobenzene (0.16 g, 0.48 mmol) were added. The reaction mixture was heated at 90 °C for 48 hours. After cooling to room temperature, the yellow precipitates were retrieved by centrifugation, washed with methanol and methylene chloride, and dried under vacuum. The powder was added to HF solution, a mixture of HF (48%, 5 mL) and water (15 mL). *Caution: the HF solution is extremely dangerous and toxic. Thus, the use of it needs special care.* The reaction mixture was stirred for 1 hour at room temperature. The powder (**H-MOP**) was washed sufficiently with a 1:1 mixture of methanol and water and dried under vacuum.

In a 50 mL Schlenk flask, **H-MOP** (50 mg) was dispersed in methanol (20 mL). The reaction mixture was heated at 70 °C for 1 hour to induce sufficiently possible swelling. Then, (NH<sub>4</sub>)<sub>2</sub>MoS<sub>4</sub> (0.114 g, 0.438 mmol) was added. The reaction mixture was heated at 70 °C for 12 hours. After cooling to room temperature, solvent was removed by rotary evaporator. The powder was dried under vacuum. Then, the powder was washed with water sufficiently until the color of the filtered water became colorless and then, dried under vacuum. The

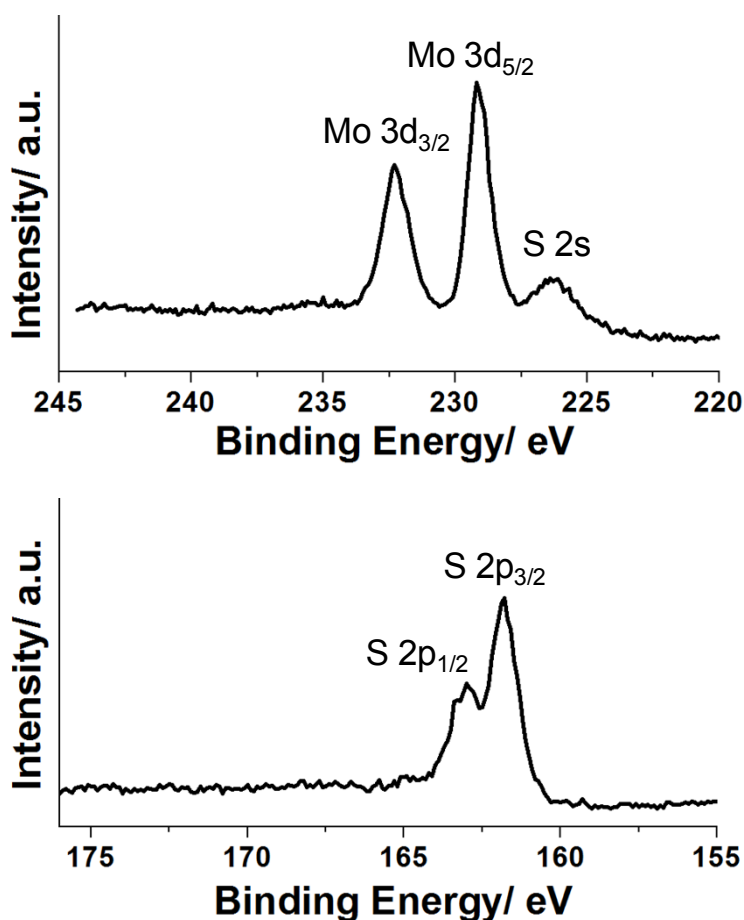
(NH<sub>4</sub>)<sub>2</sub>MoS<sub>4</sub>-loaded **H-MOP** was heated at 700 °C (the time taken for temperature increase from room temperature to 700 °C: 1 hour) for 3 hours under argon to form **MSC-2**.

For the preparation of **MSC-1**, 57 mg of (NH<sub>4</sub>)<sub>2</sub>MoS<sub>4</sub> instead of 0.114 g (NH<sub>4</sub>)<sub>2</sub>MoS<sub>4</sub> was used. The other procedure was same with that for **MSC-2**. For the preparation of **MSC-3**, 0.228 g of (NH<sub>4</sub>)<sub>2</sub>MoS<sub>4</sub> instead of 0.114 g (NH<sub>4</sub>)<sub>2</sub>MoS<sub>4</sub> was used. The other procedure was same with that for **MSC-2**.

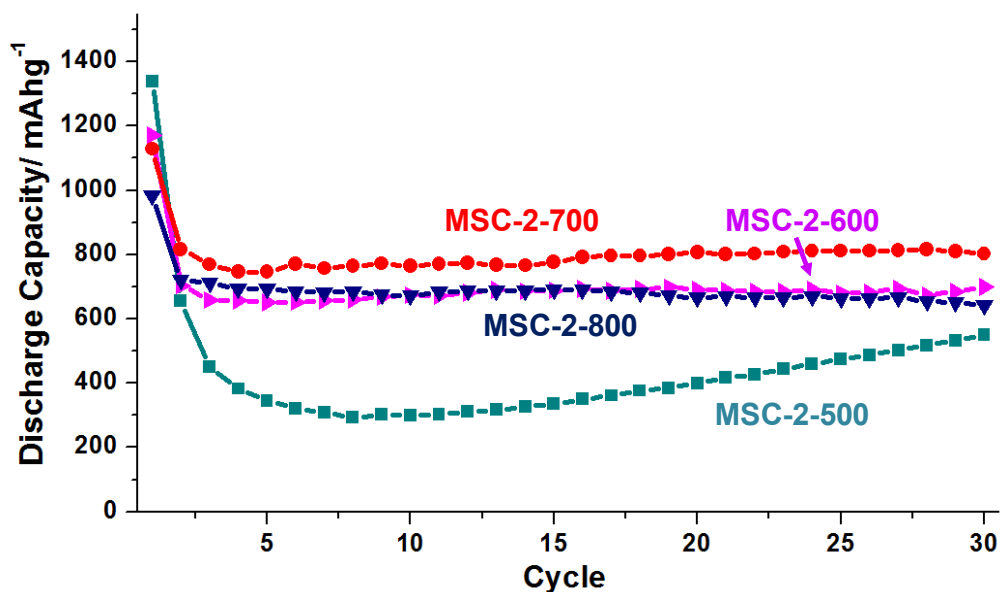
#### ***Fabrication procedure of electrochemical cells***

For the preparation of working electrodes, **MSC** nanocomposites (80 mg), polyvinylidene fluoride binder (10 mg) and Super P carbon black (10 mg) were well mixed in N-methylpyrrolidone. Cu foil was then coated with this mixture and dried under a vacuum overnight at 120 °C. The **MSC**-coated Cu electrodes were cut into a circular shape with a 1.4 cm diameter. Electrochemical cells were assembled in a glove box filled with argon. Coin-type half cells (CR2016 type) were used. Li metal was used as the counter electrode. 1 M LiPF<sub>6</sub> in ethylene carbonate/diethyl carbonate (1:1 v/v) was used as the electrolyte. The discharge/charge cycle tests were conducted using a WBCS3000 automatic battery cycler system. The cells were discharged from an open-circuit voltage to 1 mV and cycled between 1 mV and 3.0 V.

**Fig. S1** XPS spectra of **MSC-2**.



**Fig. S2** The electrochemical performance of MSC-2 which were obtained at different temperatures (500 °C, 600 °C, 700 °C, and 800 °C).



**Fig. S3** The electrochemical impedance spectroscopy (EIS) of MSC-1 ~ 3 and MSC-2 which were obtained at different temperatures (500 °C, 600 °C, 700 °C, and 800 °C).

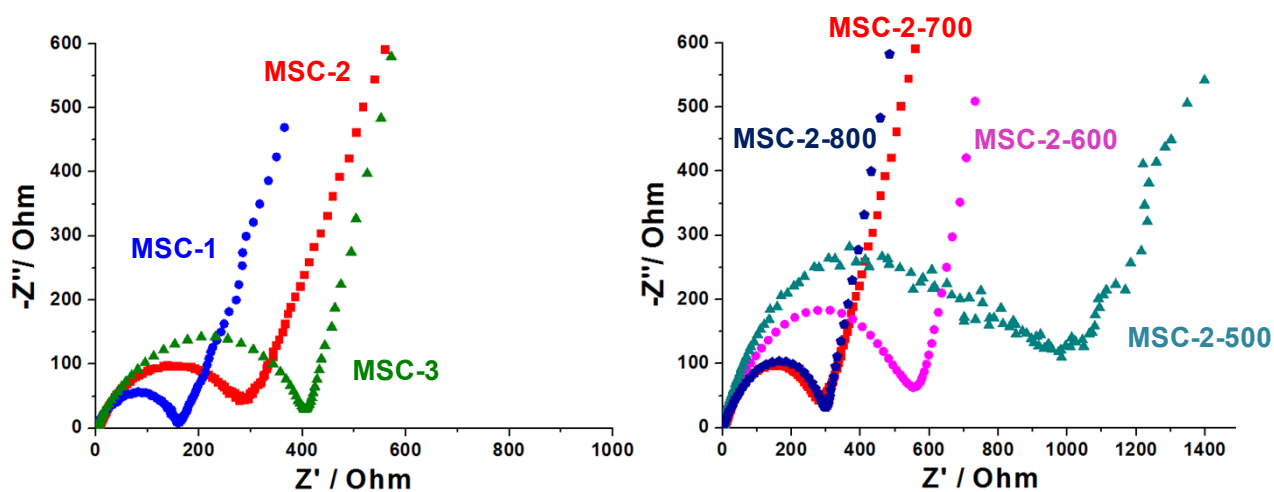


Fig. S4 Rate performance of MSC-1 ~ 3.

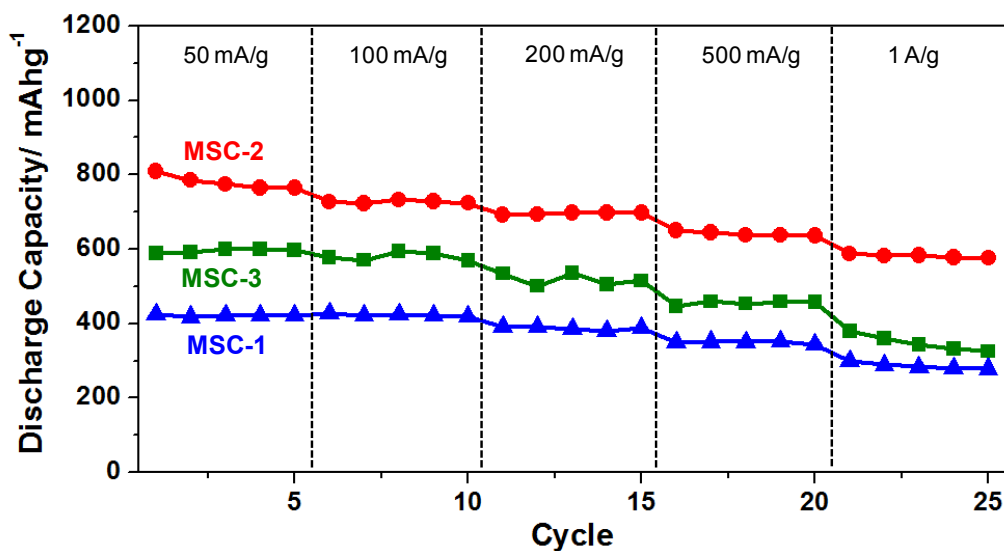
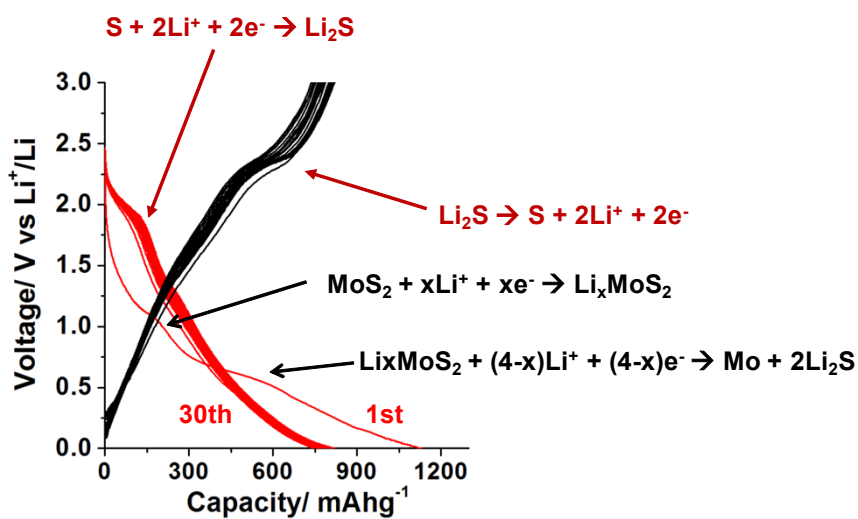
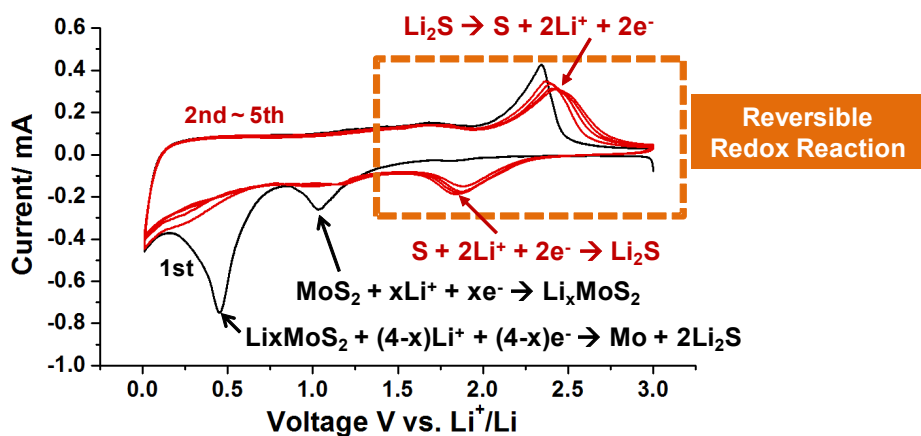
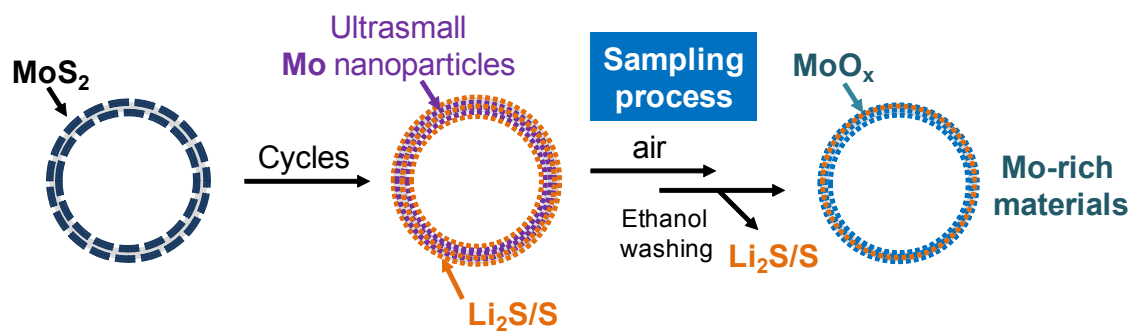


Fig. S5 Analysis of cyclic voltammograms and charge/discharge profiles of MSC-2. (Refer to ref.18 in text)

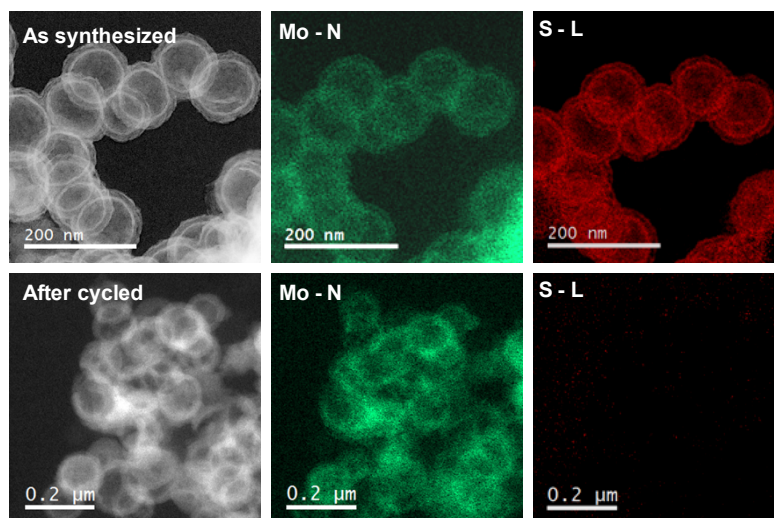


**Fig. S6** Analysis of the cycled **MSC-2**. (For the analysis of the cycled **MSC-2**, the materials were detached from Cu electrode by sonication, washed with ethanol, and dried in vacuum. During this sampling process, the  $\text{Li}_2\text{S}$  was extracted by ethanol and the ultrathin Mo particles were oxidized by air to form  $\text{MoO}_x$ .)



**Fig. S7** Elemental analysis of the cycled MSC-2 by EELS mapping and EDS spectroscopy.

### EELS mapping



### EDS analysis

