

## Supporting Information

### Well-Dispersed $O_V$ - $NiCo_2O_4$ Nanospheres Modified Separator for Li-S Batteries

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### **Preparation of cathode electrode**

The S and CNTs with a mass ratio of 4:1 were thoroughly ground and then placed in a vacuum glass tube. After drying at 155 °C for 12 hours in a blast drying oven, the mixture was taken out and mixed with Super P and PVDF at a mass ratio of 8:1:1. Magnetic stirring for 8 hours to obtain a homogeneous slurry, which was uniformly coated on a carbon coated aluminum foil and dried for 12 hours. Then cut into 12 mm disk for later use.

### **Preparation of $\text{Li}_2\text{S}_6$**

$\text{Li}_2\text{S}_6$  were obtained by adding  $\text{Li}_2\text{S}$  and S with a molar ratio of 1:5 to a solution of DOL and DME in a volume ratio of 1:1, sealed and stirring at 80 °C for 20 hours. Then diluted to a concentration of 2 mM  $\text{Li}_2\text{S}_6$  for later use.

### **The assembly of Li-S batteries**

Li-S batteries were assembled by using the  $\text{O}_V\text{-NiCo}_2\text{O}_4/\text{PP}$  as the separator, the lithium as the anode electrode and S-CNTs as the cathode electrode in an argon-filled glove box. The required electrolyte composition was 1 M LiTFSI, 0.1 M  $\text{LiNO}_3$ , and DOL and DME with a volume ratio of 1:1.

### **Material characterization**

The morphology of the materials was studied by using cold field emission scanning electron microscopy (SEM) and transmission electron microscopy (TEM). X-ray photoelectron spectroscopy (XPS) was used to analyze the valence states and composition of the elements. X-ray diffraction (XRD) was used to analyze the crystal structure of the materials. The sulfur content was measured by using Thermal gravimetric analysis (TGA), and electron paramagnetic resonance (EPR) was used to test the material defects/vacancies.

### **Electrochemical performance testing**

The cyclic voltammetry (CV) of the assembled batteries was measured by using a Shanghai Chenhua electrochemical workstation (CHI760E), with a scan voltage range of 1.7-2.8V. The assembled batteries were subjected to constant-current charge and discharge testing using a Shenzhen Neware (CT-4008-5 V 50 mA-164) instrument. The Electrochemical impedance spectroscopy (EIS) were measured by using a Shanghai Chenhua electrochemical workstation (CHI760E), with a frequency range 100 kHz - 0.01 Hz.

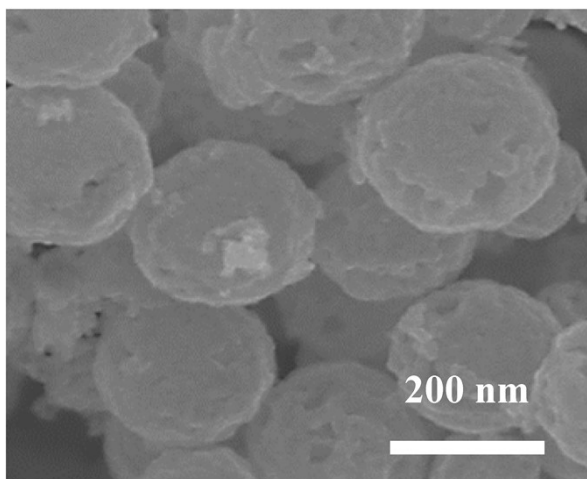
### **$\text{Li}_2\text{S}_6$ symmetric batteries**

The cathode electrode material was mixed uniformly with PVDF at a mass ratio of 9:1 and a certain amount of NMP was added to form a mixed slurry and uniformly coated on a carbon coated aluminum foil. Then dried for 12 hours cutting into 12 mm disk for later use. The Li-S batteries were assembled by using two identical electrodes as the cathode and anode electrodes, and the  $\text{O}_V\text{-NiCo}_2\text{O}_4/\text{PP}$  as the separator. The electrolyte was  $\text{Li}_2\text{S}_6$  solution (0.5 M, 30  $\mu\text{L}$ ). The obtained batteries were subjected to cyclic voltammetry testing at a scan rate of 50  $\text{mV s}^{-1}$  and a scan range of -1 V to 1 V.

### **Testing of lithium sulfide nucleation**

A battery was assembled with an electrode film as the cathode electrode, a lithium foil as the anode electrode, and the  $\text{O}_V\text{-NiCo}_2\text{O}_4/\text{PP}$  as the separator. Electrolyte containing  $\text{Li}_2\text{S}_8$  (2 M, 20  $\mu\text{L}$ ) was added to cathode electrode side while on

the anode electrode side, electrolyte without  $\text{Li}_2\text{S}_8$  (2 M, 20  $\mu\text{L}$ ) was added. The assembled battery was discharged to 2.06 V at a constant current of 0.112 mA, and subjected to constant potential testing at 2.05 V until the current dropped below  $10^{-5}$  A.



**Fig. S1.** SEM of  $O_V$ -NiCo<sub>2</sub>O<sub>4</sub> NSs

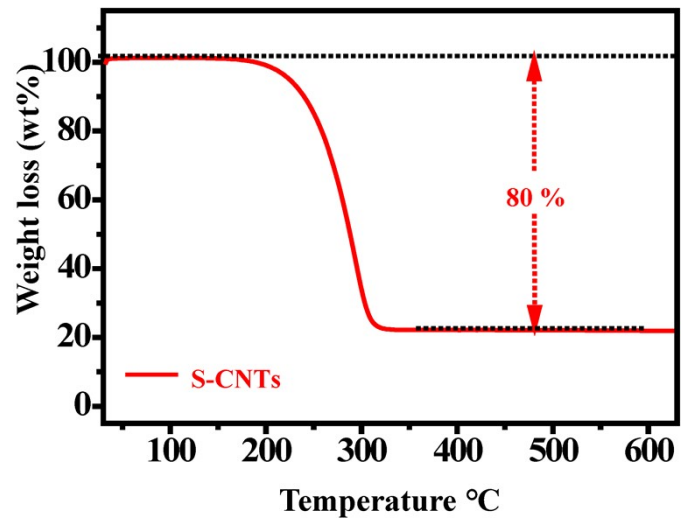
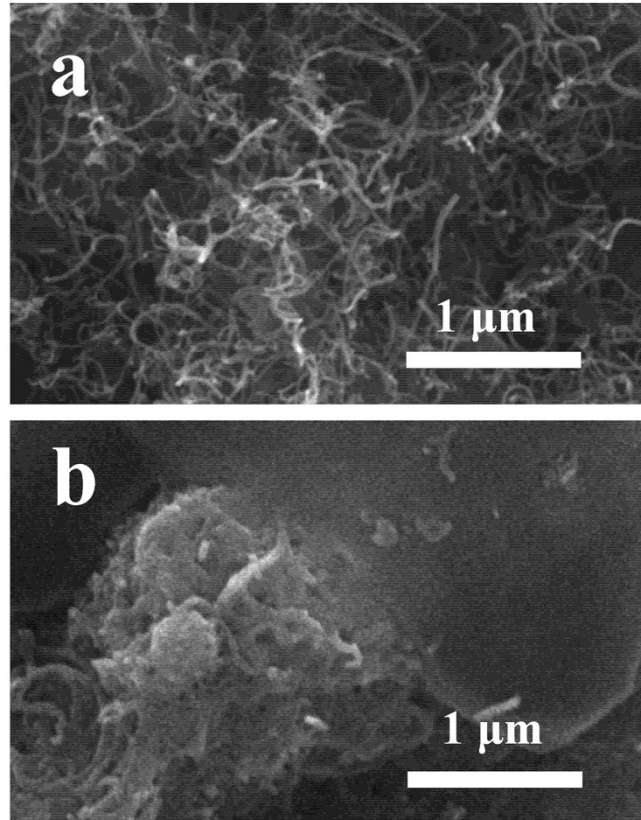
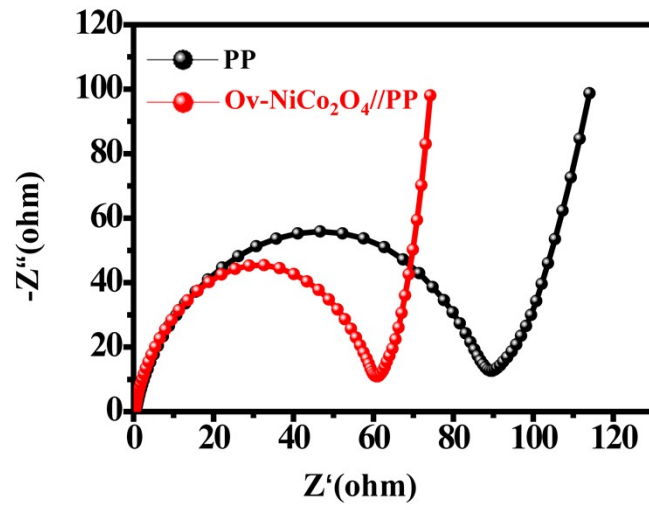


Fig. S2. TGA patterns of S-CNTs



**Fig. S3.** (a) SEM images of CNTs and (b) S-CNTs



**Fig. S4.** EIS patterns of PP and Ov-NiCo<sub>2</sub>O<sub>4</sub>//PP