Supporting Information For

Nanosheet-Assembled MoSe₂ and S-doped MoSe₂₋ₓ Nanostructures for Superior Lithium Storage Properties and Hydrogen Evolution Reactions

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Figure S1. Low-magnification SEM image of as-prepared MoSe₂ nanomaterial.

Figure S2. HRTEM image and SAED pattern of MoSe₂ nanocaterpillars. The typical SAED pattern shows concentric diffraction rings that can be indexed to the (100) and (110) diffractions of MoSe₂, indicating its polycrystalline nature.
Figure S3. (a) XRD pattern of MoSe$_2$ nanocaterpillars through vacuum drying; (b) Energy-dispersive X-ray spectroscopy spectrums of MoSe$_2$ nanocaterpillars.

Figure S4. XPS spectra of (a) Mo 3d and (b) S 2p peaks for MoSe$_2$ nanostructures.

Figure S5. Raman spectrum of MoSe$_2$ nanostructures.
Figure S6. (a) XRD pattern of S-doped MoSe$_{2-x}$ nanosheets and S-doped MoSe$_{2-x}$ nanotubes; (b) XRD pattern of MoSe$_2$ nanocaterpillars (1), S-doped MoSe$_{2-x}$ nanosheets (2), S-doped MoSe$_{2-x}$ nanotubes (3) and MoS$_2$ (4). As shown in Figure S6b, all peaks of as-prepared MoS$_2$ can be assigned to MoS$_2$ phase. It was found that increasing the sulfur content results in a continuous right shift for the diffraction (110) peaks. At the same time, the diffraction peaks at 2$\theta$ = 16° for S-doped MoSe$_{2-x}$ nanotubes can be discernible. Dotted lines are added to facilitate in tracking the shifting of (110) peak.
Figure S7. (a-b) TEM images and SAED pattern of S-doped MoSe$_{2-x}$ nanosheets. (c) HAADF-STEM image and the corresponding EDS mapping images of S-doped MoSe$_{2-x}$ nanosheets, indicating the homogeneous distribution of S, Mo and Se.
Figure S8. (a-b) Low-magnification SEM image of as-prepared S-doped MoSe$_{2-x}$ nanotubes. (c-d) TEM images and SAED pattern of S-doped MoSe$_{2-x}$ nanotubes. (e) Corresponding EDS line scan profiles.

Figure S9. Energy-dispersive X-ray spectroscopy spectrums of as-prepared S-doped MoSe$_{2-x}$ nanotubes.
Figure S10. XPS spectra of (a) S 2p peaks, Se 3d (b) and Mo 3d and S 2s peaks (c) for as-prepared S-doped MoSe$_{2-x}$ nanotubes.

Table S1. EDX analysis of the as-prepared samples.

<table>
<thead>
<tr>
<th>Atomic %</th>
<th>S-doped MoSe$_{2-x}$ nanosheets</th>
<th>S-doped MoSe$_{2-x}$ nanotubes</th>
</tr>
</thead>
<tbody>
<tr>
<td>S (K)</td>
<td>11.3</td>
<td>23.2</td>
</tr>
<tr>
<td>Mo (L)</td>
<td>45</td>
<td>51.5</td>
</tr>
<tr>
<td>Se (K)</td>
<td>43.7</td>
<td>25.3</td>
</tr>
</tbody>
</table>
Figure S11. (a) TEM image of the product in pure octylamine system. TEM images of the products with adding different alcohol: (b) oleyl alcohol; (c) heptyl alcohol; (d) alcohol.

Figure S12. (a) N\textsubscript{2} adsorption/desorption isotherms and the corresponding pore size distribution of S-doped MoSe\textsubscript{2-x} nanotubes. (b) N\textsubscript{2} adsorption/desorption isotherms and the corresponding pore size distribution of MoSe\textsubscript{2} nanocaterpillars. (c) N\textsubscript{2} adsorption/desorption isotherms and the corresponding pore size distribution of S-doped MoSe\textsubscript{2-x} nanosheets.
Figure S13. TEM image of S-doped MoSe$_{2-x}$ nanotubes after annealing.

Figure S14. The first three cycles CV curves of all samples at a scan rate of 0.1 mV s$^{-1}$. S-doped MoSe$_{2-x}$ nanotubes (a), MoSe$_2$ nanocaterpillars (b) and S-doped MoSe$_{2-x}$ nanosheets (c). For MoSe$_2$ nanocaterpillars, the reduction peak at $\sim$0.8 V during the first cycle may be attributed to the formation of Li$_x$MoSe$_2$, as shown in Figure S14b. During the following discharge cycles, a strong peak observed at $\sim$0.8 V disappears while two new peaks are repeatedly observed at $\sim$1.3 V and $\sim$1.9 V.
Figure S15. Rate performance of S-doped MoSe$_{2-x}$ nanotubes at various current densities.

Figure S16. TEM images of the S-doped MoSe$_{2-x}$ nanotubes after cycling for 30 cycles.
Figure S17. Nyquist plots of MoSe$_2$ nanocaterpillars, S-doped MoSe$_{2-x}$ nanosheets and S-doped MoSe$_{2-x}$ nanotubes.