## **Electronic Supplementary Information**

## Cyano-Bridged Coordination Polymer Hydrogel-Derived Sn–Fe Binary Oxide Nanohybrids with Structural Diversity: From 3D, 2D, to 2D/1D and Enhanced Lithium-Storage Performance

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**Fig. S1** Photographs of the Sn–Fe cyanogels with different molar ratios of Sn/Fe from 3:1 to 1:3 taken by every four hours within the initial 24 h: (a) 0 h, (b) 4 h, (c) 8 h, (d) 12 h, (e) 16 h, (f) 20 h, and (g) 24 h.



**Fig. S2** Morphological and structural features of the 2D/1D SnO<sub>2</sub>–Fe<sub>2</sub>O<sub>3</sub> hierarchitectures: (a, b) TEM images, (c) HRTEM image and its FFT pattern (inset), and (d) TEM-EDS elemental mappings of Sn (green), Fe (red), and O (purple).



Fig. S3 Morphological and structural features of the 2D/1D SnO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> hierarchitectures: (a, b) TEM images and

(c) HRTEM image and its FFT pattern (inset).



Fig. S4 Morphological and structural features of the 2D/1D SnO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> hierarchitectures: (a, b) TEM images and

(c) HRTEM image and its FFT pattern (inset).



**Fig. S5** TEM-EDS elemental mappings of the 2D SnO<sub>2</sub>–Fe<sub>2</sub>O<sub>3</sub> nanosheets.



Fig. S6 TEM images of the semi-folded (a), crumpled (b), and scrolled (c-f) 2D SnO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> nanosheets.



Fig. S7 SEM images of the 2D  $SnO_2$ -Fe<sub>2</sub>O<sub>3</sub> nanosheets.



Fig. S8 TEM-EDS elemental mappings of the 3D  $SnO_2$ -Fe<sub>2</sub>O<sub>3</sub> networks.



Fig. S9 Nitrogen adsorption/desorption isotherms (a) and pore diameter distribution from the desorption branch (b) of the  $2D/1D SnO_2$ -Fe<sub>2</sub>O<sub>3</sub> hierarchitectures.



Fig. S10 Nitrogen adsorption/desorption isotherms (a) and pore diameter distribution from the desorption branch (b)

of the 2D  $SnO_2$ -Fe<sub>2</sub>O<sub>3</sub> nanosheets.



**Fig. S11** Nitrogen adsorption/desorption isotherms (a) and pore diameter distribution from the desorption branch (b) of the 3D SnO<sub>2</sub>–Fe<sub>2</sub>O<sub>3</sub> networks.



**Fig. S12** Morphological, compositional, and structural features of the bare SnO<sub>2</sub> nanosheets: (a) TEM image, (b) EDS spectrum, and (c) XRD pattern.

Bare SnO<sub>2</sub> nanosheets has been obtained by dissolving Fe<sub>2</sub>O<sub>3</sub> component in 2D SnO<sub>2</sub>–Fe<sub>2</sub>O<sub>3</sub> nanosheets with a 6 M HCl solution (Fig. S12). As seen from the TEM image, the product retains the 2D sheet-like morphology of the 2D SnO<sub>2</sub>–Fe<sub>2</sub>O<sub>3</sub> nanosheets after the dissolution of Fe<sub>2</sub>O<sub>3</sub> component (Fig. S12a). The compositional characterization of the product has been examined by EDS. As seen in Fig. S12b, the strong peaks for Sn and O elements come from SnO<sub>2</sub> nanocrystals in the bare SnO<sub>2</sub> nanosheets, whereas the additional Cu elemental peak originates from the copper-grid used in the TEM tests. Additionally, the molar ratio of Sn/Fe is determined to be 99.98:0.02 from the EDS spectrum, indicating the complete dissolution of Fe<sub>2</sub>O<sub>3</sub> nanocrystals. Moreover, all the diffraction peaks in the XRD pattern of bare SnO<sub>2</sub> nanosheets can be indexed to tetragonal SnO<sub>2</sub> (JCPDS: 41-1445, Fig. S12c), further confirming the entire removal of Fe<sub>2</sub>O<sub>3</sub> nanocrystals in 2D SnO<sub>2</sub>–Fe<sub>2</sub>O<sub>3</sub> nanosheets.



**Fig. S13** TEM images (a, b) and XRD pattern (c) of the FeOOH nanorod precursors and TEM images (d, e) and XRD pattern (f) of the bare Fe<sub>2</sub>O<sub>3</sub> nanorods.

FeOOH nanorods was prepared by a facile hydrothermal route. Typically, 80 ml of 0.5 M FeCl<sub>3</sub> solution was transferred into a 100 mL Teflon-lined stainless steel autoclave and heated at 90 °C for 12 h, yielding the FeOOH nanorods (Fig. S13a-c). As observed, the product manifests a typical rod-like morphology (Fig. S13a, b), and the observed crystalline phase can be indexed to tetragonal FeOOH (JCPDS: 75-1594, Fig. S13c), indicating the formation of FeOOH nanorods.

The FeOOH nanorods can serve as precursors for the selective formation of iron oxide nanorods including hematite, magnetite, and maghemite under appropriate conditions. Herein, the FeOOH nanorods was annealed at  $600 \,^{\circ}$ C in air for 3 h at a heating rate of 1 K min<sup>-1</sup>. After annealing, the observed crystalline phase has transformed from tetragonal FeOOH to hexagonal Fe<sub>2</sub>O<sub>3</sub> (JCPDS: 33-0664, Fig. S13f), indicating the successful transformation from FeOOH precursor to Fe<sub>2</sub>O<sub>3</sub> product. The TEM images indicate that the oxide product inherits the 1D rod-like morphology of the FeOOH precursor (Fig. S13d, e). Moreover, the Fe<sub>2</sub>O<sub>3</sub> product exists in the form of porous structures with abundant cavities in the nanorods.



Fig. S14 Coulombic efficiencies versus cycle numbers for the 2D/1D SnO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> hierarchitectures, bare SnO<sub>2</sub> nanosheets, and bare Fe<sub>2</sub>O<sub>3</sub> nanorods.



Fig. S15 Discharge capacities versus cycle numbers for the 2D/1D SnO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> hierarchitectures in the potential range of 0.01-3 V at a current density of 100 mA g<sup>-1</sup> from 100<sup>th</sup> to 200<sup>th</sup> cycles.



Fig. S16 The  $10^{\text{th}}$ ,  $20^{\text{th}}$ ,  $50^{\text{th}}$ ,  $100^{\text{th}}$  and  $200^{\text{th}}$  discharge and charge curves for the 2D/1D SnO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> hierarchitectures.