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Electronic Supplementary Information

Pyrolysis of Cyano-Bridged Hetero-Metallic Aerogels: A General Route to Immobilize Sn–M (M=Fe, Ni) Alloys within Carbon Matrix for Stable and Fast Lithium Storage

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Fig. S1 Photographs of the glucose/Sn–Fe hydrogel (a), urea/Sn–Fe hydrogel (b), glycine/Sn–Fe suspension (c), HMTA/Sn–Fe suspension (d), and FTIR spectra of the glucose/Sn–Fe aerogel (e), urea/Sn–Fe aerogel (f), freeze-dried glycine/Sn–Fe (g) and HMTA/Sn–Fe (h) suspensions.



Fig. S2 Nitrogen adsorption/desorption isotherms (a) and pore size distribution (b) of the nanoporous Sn-Fe@C network.



Fig. S3 EDS spectrum of the nanoporous Sn–Fe@C network.



Fig. S4 XRD pattern of the nanoporous Sn–Fe@C network after TGA.

As seen from TGA curve (Fig. 5b), the weight variation of Sn–Fe@C network can be mainly attributed to the oxidation of Sn–Fe alloy and carbon components during TGA tests. The oxidation of Sn–Fe alloy leads to a weight increase, whereas the removal of carbon component leads to a weight decrease of the product. Fig. S4 shows the XRD pattern of the nanoporous Sn–Fe@C network after TGA. The observed crystalline phases from the oxidation product after TGA can be indexed to SnO₂ (JCPDS no. 41-1445) and Fe₂O₃ (JCPDS no. 33-0664). Additionally, the atomic ratio of Sn and Fe in the Sn–Fe alloy is determined to be 0.9:1 by EDS spectrum (Fig. S3), and thus the atomic ratio of SnO₂ and Fe₂O₃ in the oxidation product is 0.9:0.5. Therefore, the carbon content in the Sn–Fe@C network can be calculated based on the following equation:

$$C(\text{wt\%}) = 100 - \text{Sn-Fe}(\text{wt\%})$$

= 100 - 100 × $\frac{\text{molecular weight of (0.9 \text{Sn and Fe})}}{\text{molecular weight of (0.9 \text{SnO}_2 \text{ and 0.5 Fe}_2 \text{O}_3)}}$ × $\frac{\text{final weight of SnO}_2 \text{ and Fe}_2 \text{O}_3}{\text{initial weight of Sn-Fe@C network}}$



Fig. S5 XPS spectra of the nanoporous Sn–Fe@C network: (a) survey spectrum, and (b) Sn $3p_{3/2}$ and Fe $2p_{3/2}$ spectra.



Fig. S6 TEM images of the Sn–Fe–C composite (a, b) and CA-derived carbon (c, d).



Fig. S7 Nitrogen adsorption/desorption isotherms of the Sn-Fe-C composite.



Fig. S8 The 1st, 2nd, 10th, 20th, 50th, and 100th discharge and charge curves of the nanoporous Sn– Fe@C network in the potential range of 0.01-2 V at a current density of 100 mA g⁻¹.



Fig. S9 HRTEM image of the nanoporous Sn–Fe@C network in a fully de-lithiated state (2.0 V vs. Li⁺/Li) after 100 cycles.



Fig. S10 Nyquist plots of the nanoporous Sn–Fe@C network in comparison with Sn–Fe–C composite and CA-derived carbon in fresh cells.



Fig. S11 Photographs of the cyano-bridged Sn–Ni hydrogel (a), CA/Sn–Ni hydrogel (b), and their corresponding models (*insets*).



Fig. S12 FTIR spectra of the $K_2Ni(CN)_4$ reagent (curve *a*), cyano-bridged Sn–Ni aerogel (curve *b*), and CA/Sn–Ni aerogel (curve *c*).



Fig. S13 TEM images of the nanoporous Sn–Ni@C network (a, b) and Sn–Ni–C composite (c, d).