Supporting Information

Polymer–Inorganic Composite Protective Layer for Stable Na Metal Anodes

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Figure S1. Schematic diagram for the preparation of PSN@Cu current collectors.



Figure S2. SEM images of PSN@Cu with PVDF/Sn mass ratio of (a) 8:2, (b) 6:4, (c) 4:6 and (d) 2:8 before cycling; (e) the cycling performance of Na/Na symmetrical cells using PSN@Cu current collectors with different PVDF/Sn mass ratios with a cycling capacity of 1 mAh cm⁻² at 1 mA cm⁻².

The commercial Cu current collector is so uneven and rough that the bulge of Cu current collector could be still observed after coating PSN protective layer.

Electrochemical performances of PSN@Cu current collectors with different PVDF/Sn mass ratios (8:2, 6:4, 4:6 and 2:8) were also investigated. As shown in Figure S2a-d, more Sn particles can be observed as PVDF/Sn mass ratio decreases, and the agglomeration of Sn particles gradually appear when the PVDF/Sn mass ratio is 4:6 or 2:8. It is found that PSN@Cu current collectors present the best electrochemical performance with a PVDF/Sn mass ratio of 6:4 (Figure S2e). It is conjectured that the inferior electrochemical performance with a PVDF/Sn mass ratio of 8:2 is originated from the insufficient Sn particles, which gives rise to

poor mechanical modulus and ionic conductivity. At the same time, when the PVDF/Sn mass ratio is 4:6 or 2:8, the excess Sn particles cause agglomeration, thus leading to preferential and uneven deposition. On the other hand, the excess Sn particles also result in the decreased flexibility of PSN protective layer, which is difficult to tolerate the volume change and the interface fluctuation. Therefore, the significantly improved electrochemical performance can only be achieved in the rational combination of PVDF matrix and Sn nanoparticles.



Figure S3. Digital images of bare Cu, PVDF@Cu and PSN@Cu current collectors, respectively.



Figure S4. Cross-sectional SEM image of the PSN@Cu current collector.



Figure S5. Fitting Young's modulus value of ten different sites on PSN artificial protective layer and the average value is 5.48 GPa.



Figure S6. (a) Fitting Young's modulus value of ten different sites on PVDF and the average value is 1.79 GPa; (b) The force–distance curve and corresponding fitting Young's modulus value of the PVDF.

The Young's modulus of Cu is ~116 GPa,¹ which is much higher than the Young's modulus of the PVDF (1.79 GPa) and PSN layer (5.48 GPa).



Figure S7. First cycle voltage profiles of Cu, PVDF@Cu and PSN@Cu electrodes from 0.02 V to 2 V at 0.01 mA cm⁻².



Figure S8. Nyquist plots of bare Cu, PVDF@Cu and PSN@Cu current collectors before cycling.

The Na⁺ ion diffusion coefficients of bare Cu, PVDF@Cu and PSN@Cu current collectors can be obtained from the oblique lines in the low-frequency regions of the Nyquist plots according to the following Equations:

$$Z' = R_s + R_{ct} + \sigma \omega^{-1/2}$$
(1)

$$D = R^2 T^2 / (2C^2 F^4 S^2 \sigma^2)$$
(2)

where the Z' is the real part of impedance, R_s the Ohm resistance, R_{ct} the charge transfer resistance, σ the Warburg factor, ω the angular frequency, D the Na⁺ ion diffusion coefficient, R the gas constant, T the absolute temperature, C the molar concentration of Na⁺ ions in the electrolyte, F the Faraday's constant, and S the surface area of the electrodes. As shown in Figure 2b, the Z' has a linear relationship with $\omega^{-1/2}$ and σ is determined by the slope of the lines, and thus D could be obtained ($D_{Cu}=1.53 \times 10^{-14}$ cm² S⁻¹, $D_{PVDF}=3.14 \times 10^{-14}$ cm² S⁻¹, $D_{PSN}=6.12 \times 10^{-13}$ cm² S⁻¹).



Figure S9 SEM images of (a) bare Cu current collector, (b) PVDF@Cu current collector and

(c) PSN@Cu current collectors after depositing 4 mAh cm^{-2} of Na.



Figure S10 The initial CEs of Na plating/stripping on bare Cu, PVDF@Cu and PSN@Cu current collectors at 2 mA cm⁻² with (a) a cycling capacity of 1 mAh cm⁻² and (b) a cycling capacity of 2 mAh cm⁻².

Before the CE test, these batteries were initially cycled for 5 times from 0.01 to 1 V at 0.4 mA cm⁻² to remove surface contaminations (Figure S10).

With cycling capacity of 1 mAh cm⁻², the CE of bare Cu current collector suffers from drastic oscillation from initial cycles probably due to the formation of unstable SEI (Figure S10a), which agrees with the previous reports². When cycling capacity increases to 2 mAh cm⁻², the severely fluctuating CEs are observed on both bare Cu current collectors and PVDF@Cu current collectors from the initial cycle, (Figure S10b). Notably, reversible and stable CEs are achieved on PSN@Cu current collectors after 10 cycles with cycling capacity of 1 mAh cm⁻² and 2 mAh cm⁻², demonstrating the effectiveness of PSN coating as protective layer.



Figure S11. The detailed plating/stripping voltage curves of (a) bare Cu current collectors, (b) PVDF@Cu current collectors and (c) PSN@Cu current collectors with a cycling capacity of 1 mAh cm⁻² at 2 mA cm⁻².



Figure S12. The detailed plating/stripping voltage curves of (a) bare Cu current collectors, (b) PVDF@Cu current collectors and (c) PSN@Cu current collectors with a cycling capacity of 2 mAh cm⁻² at 2 mA cm⁻².



Figure S13. The cycling performance of Na/Na symmetrical cells using bare Cu and PSN@Cu current collectors with a cycling capacity of 1 mAh cm⁻² at 2 mA cm⁻².

Bare Cu current collector only survives for ~130 h with a significantly increased overpotential, while PSN@Cu current collector illustrates prolonged cycling lifetime of 1200 h with a stable Na plating/stripping process.



Figure S14. The cycling performance of Na/Na symmetrical cells using bare Cu and PSN@Cu current collectors with a cycling capacity of 2 mAh cm⁻² at 2 mA cm⁻².

Bare Cu current collector fails quickly at ~300 h with large overpotential, while PSN@Cu current collector presents prolonged cycling lifetime of 750 h with a stable Na plating/stripping process.



Figure S15. The cycling performances of Na/Na symmetrical cells using PVDF@Cu current collectors.



Figure S16. The cycling performance of Na/Na symmetrical cells using bare Cu and PSN@Cu current collectors with a cycling capacity of 2 mAh cm⁻² at 10 mA cm⁻².



Figure S17. XPS spectra of the SEI formed on PSN@Cu current collectors after 80 cycles with a cycling capacity of 1 mAh cm⁻² at 2 mA cm⁻², the corresponding Sn 3d, Na 1s, P 2p and Cu 2p spectra and sputtering time are shown in rows and columns.

At 0 s of sputtering, the C 1s spectrum at ~284.8 eV, ~286.6 eV and ~287.9 eV are assigned to C-C, C-O and O-C=O,³ the O 1s spectrum can be deconvoluted into four peaks at ~530.7 eV (Na₂O),⁴ ~532.5 eV (Na_{2-x}O₂)⁴ ~531.9 eV (C-O)⁵ and ~533.3eV (O-C=O).⁶ The F 1s spectrum presents one peak at ~684 eV (Na-F)^{7,8}, and peaks at ~1070.8 eV⁹ and ~1072.2 eV¹⁰ in Na 1s spectrum belong to Na-F and Na-O, respectively. After sputtering for 40 s and 80 s, organic RCOONa⁵ is disappeared. The content of P is most nothing during whole sputtering.



Figure S18. Cross-sectional SEM image of the PSN@Cu current collector with deposited Na (1 mAh cm⁻²).

From cross-sectional SEM image of the PSN@Cu current collector, it is found that PSN protective layer is located on the top layer, and Na is deposited underneath the PSN layer.



Figure S19. XPS spectra of the SEI formed on PSN@Cu current collectors after 80 cycles with a cycling capacity of 1 mAh cm⁻² at 2 mA cm⁻², the corresponding C 1s, O 1s, F 1s, P 2p, Na 1s and Cu 2p spectra and the sputtering time are shown in rows and columns.

Before sputtering, the C 1s spectrum can be fitted using three peaks with binding energies of ~284.8 eV (C-C), ~286.8 eV (C-O) and ~288.8 eV (O-C(=O)O),^{11,12} and peaks at ~529.8 eV, ~531.2 eV and ~533.2 eV in the O 1s spectrum are assigned to Na-O, C-O and O-P=O/O-C(=O)O.^{3,5} F 1s spectrum shows two peaks at ~683.6 eV (Na-F) and ~687 eV (P-F),^{7,8} and peaks at ~132.2 and ~137 eV in the P 2p spectrum are attributed to Na_xPF_yO_z and NaPF₆⁵, respectively. Na 1s spectrum presents two peaks at ~1070.8 eV⁹ and ~1072.2 eV¹⁰ belonging to Na-F and Na-O, respectively. After sputtering for 40, 80, 120 and 180, other peaks still exist

except the absence of the C-O peak compared with the top surface of SEI. Therefore, the SEI of Cu current collector is mainly made up of RCH₂ONa, ROCO₂Na, Na₂O, NaF, NaPF₆ and $Na_x PF_y O_z$.

The peak of Cu was observed after sputtering for 120 s and showed the content of $\sim 8\%$ after sputtering for 180 s. 180 s of sputtering is corresponding to SEI thickness (13.7 nm) in view of sputtering rate (~ 4.58 nm/min), indicating that the SEI thickness of Cu current collector is significantly higher than 13.7 nm according to the estimation method of previous work.



Figure S20. XPS atomic concentration of various elements as a function of sputtering time for the SEI formed on (a) PSN@Cu current collectors and (b) bare Cu current collectors.



Figure S21. Cycling performance of full cells using bare Cu current collectors and PSN@Cu

current collectors at current density of 0.2 A g^{-1} .



Figure S22. Voltage profiles of full cells using (a) bare Cu current collectors and (b) PSN@Cu current collectors at current density of 0.2 A g^{-1} .

Table S1. Sumr	mary of cycle performar	nce of Na plating/stripping	g behaviors. (The electrolyte
concentration is	1 M unless other specif	ied)	
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Modified approach	Current density [mA cm ⁻²]	Cycle	Cycling time [h]	Depth of discharge	Electrolyte	Ref.
3D wettable carbon fiber	0.5	75 300		l mAh cm ⁻² of Na foil (low)	NaClO₄ in EC/DMC/EMC with 5% FEC	13
	1	120	480	2	NaClO ₄ in EC/PC	14
3D flexible carbon felt	3	~120	~160	2 mAn cm ⁻ or Na		
	5	~125	~100	1011 (10w)		
Sodium-alloy-fluoride	0.5	600	600	0.25 mAh cm ⁻² of Na foil (low)	NaClO ₄ in EC/PC with 2% FEC	15
	0.5	105	500	1 mAh cm ⁻² of Na	NaClO ₄ in EC/PC	16
SnCl ₂ -added electrolyte		125	500	foil (low)	(with SnCl ₂)	
CuNW-Cu 1		~260	~520	1 mAh cm ⁻² of 2 mAh cm ⁻² (50 %)	NaPF ₆ in diglyme	17
Chemically engineered	0.5	100	400	1 mAh cm ⁻² of Na	NaClO ₄ in	18
copper	1	100	200	foil (low)	EC/DEC	
Cash an ann an	0.5 62	(2	250	1 mAh cm ⁻² of Na	NaSO ₃ CF ₃ in	19
Carbon paper		62		foil (low)	diglyme	
2D contrast folk	1	550	1100	1 mAh cm ⁻² of 10	N-DE in dislama	20
3D carbon leit	2	350 350		mAh cm ⁻² (10 %)	NaPF ₆ in digiyme	20
No S. additivos	2	200	400	1 mAh cm ⁻² of Na	NaPF ₆ in diglyme	5
	2	200	400	foil (low)	(with Na ₂ S ₆)	<i></i>
Ag nanowire-based	1	400	800	$1 \text{ mAh cm}^{-2} \text{ of } 4$	1.5 M NaSO ₃ CF ₃	21
nanopaper	1	400	800	mAh cm ⁻² (25%)	in diglyme	
	2	200	122	1 mAh cm ⁻² of 3		
MV	3	~200	~133	mAh cm ⁻² (33.3%)	N-DE in development	2
MXene	3	300	600	3 mAh cm ⁻² of 5	- NaPF ₆ in dyglyme	
	5	200	240	mAh cm ⁻² (60%)		
NSCNT paper interlayer	1	250	500	1 mAh cm ⁻² of Na	NaSO ₃ CF ₃ in	22
		250	500	foil (low)	diglyme	
Na ONaDO 2	1	135	270	1 mAh cm ⁻² of Na		23
Na@NaPS-3	3	210	140	foil (low)	Napp ₆ in EC/PC	
	1	1150	2300	1 mAh cm ⁻² of 4	NoDE in dislume	
DEN@C	2	1200	1200	mAh cm ⁻² (25%)		This
r sm@Cu	1	625	2500	2 mAh cm ⁻² of 4	TVAT F6 III UIGIYINE	work
	2	375	750	mAh cm ⁻² (50%)		

Modified approach	Current density & Capacity	Cycle	Cycling time (h)	Coulombic efficiency	Electrolyte	Ref.
CuNW-Cu	2 mAh cm ⁻¹ at 1 mA cm ⁻²	350	1400	99.7%	NaPF ₆ in diglyme	17
MXene	4 mAh cm ⁻¹ at 3 mA cm ⁻²	200	300	99.1%	NaPF ₆ in diglyme	2
	4 mAh cm ⁻¹ at 4 mA cm ⁻²	250	500	98.9%		
	5 mAh cm ⁻¹ at 5 mA cm ⁻²	100	200	98.5%		
NSCNT paper interlayer	1 mAh cm ⁻¹ at 1 mA cm ⁻²	200	400	99.8%	NaSO ₃ CF ₃ in diglyme	22
3D Ni@Cu	$0.5 \text{ mAh cm}^{-1} \text{ at } 1 \text{ mA cm}^{-2}$	220	220	99.5%	NaPF ₆ in DEG/DME	24
3D carbon felt	1 mAh cm ⁻¹ at 0.5 mA cm ⁻²	500	2000	99.3%	NaPF ₆ in diglyme	20
Cu@Au	1 mAh cm ⁻¹ at 1 mA cm ⁻²	150	300	99.8%	NaSO ₃ CF ₃ in	25
	1 mAh cm ⁻¹ at 2 mA cm ⁻²	300	300	99.8%	diglyme	
Cu@Sn-NPs	1 mAh cm ⁻¹ at 2 mA cm ⁻² 2 mAh cm ⁻¹ at 2 mA cm ⁻²	2000 600	2000 1200	99.9% 99.8%	NaOTf in diglyme with NaTFSI	26
Cu@Sb-MPs	1 mAh cm ⁻¹ at 2 mA cm ⁻² 2 mAh cm ⁻¹ at 2 mA cm ⁻²	1700 600	1700 1200	99.9% 99.9%	NaOTf in diglyme with NaTFSI	26
PSN@Cu	1 mAh cm ⁻¹ at 2 mA cm ⁻²	880	880	99.8%	NaPF ₆ in	This
	2 mAh cm ⁻¹ at 2 mA cm ⁻²	1400	2800	99.7%	diglyme	work

 Table S2. Summary of CE performance of Na plating/stripping behaviors. (The electrolyte concentration is 1M unless other specified)

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