Electronic Supplementary Information (ESI) for

An anode-less, mechanical-flexible and energy/power dense sodium battery prototype

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Fig. S1 (a) TGA-DSC plots of dried gel at a heating rate of 10 °C min⁻¹. (b) Cu₂NiZn particle size statistics in Cu₂NiZn@CNT. (c) TEM, (d) HRTEM images of the Cu₂NiZn particle and (e) corresponding histogram.

In order to evaluate the temperature at which combustion occurs, thermogravimetric analysis (TGA)differential scanning calorimetry (DSC) analysis was performed (Fig. S1a). Two small endothermic peaks at 135 °C and 170 °C indicate the release of H_2O molecules attached to the dried gel. A strong exothermic peak with a sharp decrease in weight was observed at 235 °C, at which temperature combustion occurred.

a	Cu	b (111)	c Atom	Radius (pm)
10 <u>n</u> m			Cu	128
NI	Zn		Ni	163
_			Zn	139
	1	7 18 19 20 21 2 2θ (degree)	2	

Fig. S2 (a) HAADF-STEM and corresponding STEM-EDS element maps of Cu, Ni and Zn of the Cu₂NiZn@CNT. (b) zooming in the (111) peak of the XRD pattern. (c) Atomic radius of Cu, Ni and Zn of Cu₂NiZn@CNT.

We zoomed in the (111) peak in the XRD pattern, and the results revealed that the peak of CuNi is shifted to a lower angle compared to the standard peak of Cu, which means that the host Cu lattice is doped with a larger radius atom Ni, expanding the crystal cell parameters and the crystal plane spacing (Fig. S2b). As Ni is replaced by smaller Zn atoms, the peak of Cu₂NiZn is shifted to a higher angle compared to the CuNi peak, and the crystal cell parameters decrease on the contrary.



Fig. S3 (a) FESEM images and (b) according enlarged image of Cu₂NiZn@CNT-15. (c) FESEM images and (d) according enlarged image of Cu₂NiZn@CNT-60.



Fig. S4 SEM of $Cu_2NiZn@CNT$ with the CVD process extended to 60 min.

As the CVD process proceeded from 30 to 60 minutes, we can see that the metal particles are either entirely encapsulated in the carbon tube or located within one end of the tube (Fig. S4).



Fig. S5 (a) Raman spectra of Cu₂NiZn@CNT-15, Cu₂NiZn@CNT and Cu₂NiZn@CNT-60. (b) TGA spectrum in the air at a heating rate of 10 $^{\circ}$ C min⁻¹, and (c) N₂ adsorption and desorption isotherms of Cu₂NiZn@CNT.



Fig. S6 (a) HAADF-STEM images and corresponding STEM-EDS element maps of the Cu₂NiZn@CNT at the alloying state. (b) TEM and HRTEM of Cu₂NiZn@CNT electrode after 10 cycles.



Fig. S7 FESEM and corresponding EDS elemental maps of the $Cu_2NiZn@CNT$ after dealloying.



Fig. S8 Atomic configurations of Na atom adsorbed (a) on Zn (001), Cu (111), Al (111), Ni (111). (b) Theoretical calculations of adsorption binding energies between different numbers of Zn atoms and CNT, Zn_1/CNT , Zn_4/CNT and Zn_{14}/CNT . (c) CV curves of Na||Cu₂NiZn@CNT at a scan rate of 0.1 mV s⁻¹ for the initial three cycles.

Based on the cyclic voltammetry (CV) analysis, the redox peaks at 0.75 V/0.73 V are attributed to the reversible Na alloying/dealloying process with Zn, subsequently, the -0.02 V/0.04 V peaks can be attributed to the deposition/stripping process of Na (Fig. S8c).^{1, 2}



Fig. S9 dQ/dV curves of Na||Cu₂NiZn@CNT and Na||CuNi@CNT batteries.

The dQ/dV curves display redox peaks almost in agreement with CV, the redox peaks at ~0.77 V/0.71 V are attributed to the reversible Na alloying/dealloying process with Zn, subsequently (Fig. S9).



Fig. S10 Binary phase diagram of Na-Zn alloy.³



Fig. S11 CV curves at a scan rate of 0.1 mV s⁻¹ for the initial three cycles of CuNi@CNT.

The overlapped CV curves and stable redox peaks demonstrate the superior electrochemical stability of active sites. For comparison, the as-synthesized CuNi@CNT above 0 V is electrochemically inert, and no redox peak can be found in CV curves of the Na||CuNi@CNT (Fig. S11).



Fig.S12 (a) Nucleation overpotentials of the Cu₂NiZn@CNT, CuNi@CNT and Al foil substrates at 1 mA cm⁻². (b) Na nucleation and deposition overpotentials measured at 1 mA cm⁻² for the Cu₂NiZn@CNT, CuNi@CNT and Al foil substrates.

Three identical cells for each substrate were assembled and measured to obtain the error bars. Thus, this effective mitigation of the nucleation barrier is highly expected to enable the regulated metallic embryo growth and deposit propagation within the scaffold.



Fig. S13 HAADF-STEM and corresponding STEM-EDS element maps of the Cu₂NiZn@CNT upon the initial deposition (0.5 mA h cm^{-2}).



Fig. S14 FESEM images and EDS elemental maps of Cu₂NiZn@CNT after stripping.



Fig. S15 (a) TEM and HRTEM images, (b) HAADF-STEM and corresponding STEM-EDS element maps of the Cu₂NiZn@CNT after 100 cycles.



Fig. S16 (a) TEM, (b) HRTEM image, (c) Enlarged view of the selected area and (d) SAED of the Cu₂NiZn particle after 100 cycles

at dealloying state.



Fig. S17 XPS spectra of Cu2NiZn@CNT electrode after 10 cycles and 100 cycles in 1 M NaPF₆/diglyme: C 1s, O 1s, Na 1s, and F1s.



Fig. S18 Photographs of the $Cu_2NiZn@CNT$ film before and after calendering process.

Benefiting from the excellent toughening of CNTs, the as-prepared $Cu_2NiZn@CNT$ film remained intact without any damage after calendering process. The results show that the composite substrate has favorable mechanical behavior and flexibility.



Fig. S19 Cross-sectional FESEM image and corresponding EDS elemental maps of C, Al and O elements AIO_x-PE separator.



Fig. S20 Contact angles of ether electrolyte with (a) AlO_x-PE and (b) bare PE membrane, respectively.



Fig. S21 (a) Nyquist plots of the coin cells (stainless steel/separator/stainless steel) for the liquid electrolyte-soaked AlOx-PE separator and PE separator. (b) The tensile properties of the PE and AlOx-PE separator.

Notably, The AlO_x-PE was obtained by spin-coating on PE membrane with AlO_x colloids, as described in the experimental section. The FESEM and corresponding elemental maps show that interconnected submicron spindlelike AlO_x particles are intimately cast on the commercial PE separator (Fig. S19). Besides, the contact angle of ether electrolyte is about 11.7° on the commercial PE and almost 0° on the AlO_x-PE separator, indicating excellent wettability of the separator and enhanced affinity with the electrolyte (Fig. S20). Beyond that, it can be calculated from the Nyquist plots in Fig. S21a, the AlO_x-PE separator displays an electrical conductivity of 0.38 S cm⁻¹, almost 5 times higher than that of a PE separator (0.07 S cm⁻¹). On the other hand, the comparison of elongation of AlO_x-PE (180%) and PE (125%) separator is also shown in Fig. S21b. Collectively from Fig. S19-Fig. S21, the modified AlO_x-PE separator can achieve mechanical strength, high ionic conductivity as well as intimate electrode integration, guaranteeing the optimal choice of the flexible substrate.



Fig. S22 Voltage profiles of CuNi@CNT at 1 mA cm⁻² with 10 mA h cm⁻².



Fig. S23 (a) SEM of CuNi@CNT electrode at stripping state after 200 cycles. (b) Aqueous solution of Cu₂NiZn@CNT and CuNi@CNT electrodes at stripping state after 200 cycles.

We qualitatively compared the amount of accumulated dead Na retained after 200 cycles of the Cu₂NiZn@CNT and CuNi@CNT electrodes based on the reaction $2Na + 2H_2O = 2NaOH + H_2 \uparrow$. Since trace gases are difficult to be detected precisely under our existing experimental conditions, we performed the comparison of Na⁰ content by testing the pH value of aqueous NaOH solutions (Fig. S23b). The detailed experimental process was as follows: firstly, Na||Cu₂NiZn@CNT and Na||CuNi@CNT button batteries were assembled with 1 M NaPF₆/diglyme electrolyte and depositing-stripping 200 cycles on the Neware battery tester (1 mA h cm⁻², 1 mA h cm⁻²), then the batteries were disassembled in the stripping state and the electrodes were cleaned with ethylene glycol dimethyl ether (DOL) organic solution. After drying under a vacuum atmosphere, the electrodes were transferred to a glass vial and 10 mL of deionizing water was added quickly. After a full reaction, the pH value of the aqueous solution was tested. The results showed that the pH values of aqueous solutions of Cu₂NiZn@CNT and CuNi@CNT electrodes.

Samples	R _e (Ω)	R _{inter} (Ω)	R _{ct} (Ω)	W ₀ (Ω)
CuNi@CNT 1 st	8.6	25.2	86.4	17.1
CuNi@CNT 100 th	7.8	10.6	24.7	8.9
Cu_NiZn@CNT 1 st	10.8	12.3	32.6	15.3
Cu ₂ NiZn@CNT 100 th	7.1	1.8	8.5	4.6

Table S1. Prices of the main chemical reagents required for the preparation of the substrate

The Cu₂NiZn@CNT electrode shows a slight decrease in interfacial resistance at the SEI after cycling ($\approx 1.8 \Omega$ after 100 cycles) compared to the initial value ($\approx 12.3 \Omega$), indicating a better stability of the SEI. The Cu₂NiZn@CNT electrode also yields a much lower SEI resistance combined with charge transfer resistance ($\approx 10.3 \Omega$) than that of the CuNi@CNT ($\approx 35.3 \Omega$) after cycling, which illustrates that better Na stripping/plating kinetics is achieved for Cu₂NiZn@CNT.



Fig. S24 (a) Tafel plots and (b) comparison of exchange current densities (I₀) of Cu₂NiZn@CNT and bare Na.



Fig. S25 (a) First cycle of galvanostatic charge/discharge voltage curves and (b) CE in 30 cycles of different full cell configurations of Na/Cu₂NiZn@CNT||NaVPO₄F, Cu₂NiZn@CNT||NaVPO₄F, Cu₂NiZn@CNT||NaVPO₄F, Al||NaVPO₄F, hard carbon||NaVPO₄F, and Na||NaVPO₄F.

We present the charge/discharge curves for the first circle of the full cell: anode-free $Cu_2NiZn@CNT||NaVPO_4F$ and $Al||NaVPO_4F$, as well as $Na/Cu_2NiZn@CNT||NaVPO_4F$, hard carbon||NaVPO_4F and $Na||NaVPO_4F$, and the initial Coulomb efficiencies (ICE) were 62.6%, 40.8%, 97.7%, 85.7% and 90.5%, respectively.

The theoretical capacity retention (CR = (CE)ⁿ) of the full cell was calculated with CE as 16.9%, 13.5%, 90.7%, 58.2% and 69.5% in 30 cycles. The results demonstrated that though the capacity retention of $Cu_2NiZn@CNT||NaVPO_4F$ is higher than that of anode-free model equipped with Al foil, it still cannot ensure the stable cycle life when employing the $Cu_2NiZn@CNT$ substrate as anode free substrate. Note that the actual capacity retention of the $Na/Cu_2NiZn@CNT||NaVPO_4F$ is higher than the theoretical calculation due to the available extra Na source.

11 Na	12 Mg								13 Al	14 Si		Excellent solubility with NaPoor solubility with Na			
19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	Used for HEAs
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Catalyst for CNT
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	Fe group metals Pt group metals Coin metals
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	
55	56	57-	72	73	74	75	76	77	78	79	80	81	82	83	Oxide formation metals
Cs	Ba	71	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Ti	Pb	Bi	

Fig. S26 Metals in the periodic table that are soluble with Na and commonly used in HEAs, and types of metal catalysts for the CNT growth.

The developed HEAs system can be broadly divided into two categories: One is the alloy system based on Al and the IV period elements Fe, Co, Ni, Cr, Cu, Mn, Ti; another one is the refractory HEAs system dominated by Mo, Ti, V, Nb, Hf, Ta, Cr and W (Fig. S26).^{4, 5} Moreover, based on binary phase diagrams, typically, elements in the periodic table with red color (i.e., Mg, Sn, Sb, Ge, In, etc.) could react with the Na, leading to the improved deposition affinity. As the traditional catalysts for the CNT growth, Fe, Co, Ni and their alloys exhibit moderate carbon solubility and high carbon diffusion rate.⁶



Fig. S27 HAADF-STEM and corresponding STEM-EDS element maps of the FeCoNiAlZn@CNT.



Fig. S28 (a) TGA spectrum, (b) XRD pattern of the of FeCoNiAlZn@CNT composite.



Fig. S29 (a) First three CV curves at 0.1 mV s⁻¹ of FeCoNiAlZn@CNT. (b) Nucleation overpotentials of the FeCoNiAlZn@CNT substrates at 2 mA cm⁻² and 2 mA cm⁻². (c) Voltage-time curves of the FeCoNiAlZn@CNT symmetric cells at step-increased current densities.



Fig. S30 FESEM image of the (a) FeCoNiZn@CNT, and (b) with Na deposition capacity of 10 mA h cm⁻² at 0.5 mA cm⁻². (c) Galvanostatic cycling voltage profiles of FeCoNiZn@CNT anode in symmetric cells at 2 mA cm⁻² with a capacity of 2 mA h cm⁻².

Tetranary alloy of FeCoNiZn was also adopted to prepare the substrate (FeCoNiZn@CNT) as shown in Fig. S30a, where the FeCoNiZn alloy particles were slightly larger than FeCoNiAlZn due to the lack of inert element Al, and the correspondingly induced CNTs were more robust. After electrochemical plating with an areal capacity of up to 10 mA h cm⁻², the anode surface remained flat without observable dendrites, revealing the favorable dendrite free behavior (Fig. S30b). Noticeably, the symmetrical cell of FeCoNiZn@CNT achieved stable cycling over 1000 h with a much lower overpotential (40 mV) at current density of 2 mA cm⁻² and deposition areal capacity of 2 mA h cm⁻² (Fig. S30c).

	Based on active materials							
C-rate	Specific capacity	Energy density	Power density					
	(mA h g⁻¹)	(W h kg ⁻¹)	(W kg ⁻¹)					
0.5	103.4	351.6	175.8					
1	102.1	347.1	347.1					
2	100.6	342.0	684.1					
3	99.5	338.3	1014.9					
4	98.2	333.9	1335.5					

 $\textbf{Table S2.} The calculation results of the specific capacity, energy density and power density of the Cu_2NiZn@CNT||NaVPO_4F pouch and power density of the Cu_2NiZn@CNT||NaVPO_4F power density and powe$ cell at different C-rates (based on the total mass of the active materials).

Concerning gravimetric energy density, which can be calculated by following equations: Energy density (W h kg⁻¹) = specific capacity (mA h g⁻¹) × average voltage (V) (1) Specific power density (W kg⁻¹) = energy density (W h kg⁻¹) × C rate (h⁻¹) (2)

Anode	Voltage hysteresis /nucleation	Full cell	Electrolyte	Separator	Prestored Na (mA h cm ⁻²)	N/P ratio	Energy density (W h kg ⁻¹)/	Citation
	overpotential						(W kg ⁻¹)	
	(mv)				1		454/24	
Sb@110 _{2-x}	/	Sb@TiO _{2-x} []	1 M NaCIO ₄ /EC:DEC	glass	/	/	151/21	57
		Na ₃ V ₂ (PO ₄) ₃ -C		microfiber				
Porous carbon-	/	Fe O.I.I		(GF)	1	1	77/125	58
Fe ₂ O ₄	/	Na ₂ FeP ₂ O ₇	1 W Ndelo4/1 C 2/01 LC	G	7	-	777125	50
(PC-Fe ₂ O ₄)								
Hollow and	45 (1 mA cm ⁻²)	Na/HpCNF	50 μL	GF	4	12.5	370/150	59
mesoporous carbon	- (-)	Na ₃ V ₂ (PO ₄) ₂ F ₃	1 M NaClO ₄ /EC:PC+5%				,	
nanofiber (HpCNF)		5 21 472 5	FEC					
Oxygen-doped	35 (1 mA cm ⁻²)/	Na/OCF	35 μL	Celgard	1	59	295/220	S10
carbon foam (OCF)	9 (0.5 mA cm ⁻²)	$Na_3V_2(PO_4)_3$	1 M NaPF ₆ /diglyme	2400				
Carbonised lignin	45 (2 mA cm ⁻²)	L700 Prussian	200 µL	GF	0	0	170/60	S11
(L700)		blue	1 M NaCF ₃ SO ₃ / diglyme					
C@Sb	31 (1 mA cm ⁻²)/	Na/C@Sb	80 μL	GF	6	25	90/75	S12
	4.3(1 mA cm ⁻²)	$Na_3V_2(PO_4)_3$	1 M NaClO ₄ /PC+5% FEC					
Oxygen-containing	10 (10 mA cm ⁻²)/	O-CCF	50 μL	Celgard	0	0	250/110	S13
carbonized coconut	9 (1 mA cm ⁻²)	$Na_3V_2(PO_4)_3$	1 M NaPF ₆ /diglyme	2400				
framework (O-CCF)								
Artificial graphite	/	Na/Graphite	1 M NaPF ₆ in DEGDME	Celgard	0.5	0.3	245/150	S14
	15 (2 mA cm ⁻²)	$Na_4Fe_3(PO_4)_2(P_2$		2300				
		O ₇)						
3D nitrogen-doped	/	Na/NCF	1 M NaClO ₄ /EC:DMC	GF	8	27.3	107/107	S15
carbon fiber (NCF)	6 (0.25 mA cm ⁻²)	$Na_3V_2(PO_4)_3$	+5% FEC					
Graphitized carbon	32 (0.5 mA cm ⁻²)/	Na/GCMs	1 M NaPF ₆ /diglyme	Celgard	4	40	98/49	S16
microspheres	20 (1 mA cm ⁻²)	O ₃ -						
(GCMs)		NaNi _{0.5} Mn _{0.2} Ti _{0.}						
		₃ O ₂						
(3D) porous Cu	20 (1 mA cm ⁻²)/	Na/PCu	1 M NaPF ₆ /DEGDME	Celgard	1	9.1	202/202	S17
PCu		$Na_3V_2(PO_4)_3$						
Na with SiO ₂ coating	5 (1 mA cm ⁻²)/	Na@SiO ₂	50 μL	Celgard	/	/	/	S18
(Na@SiO ₂)		Na ₃ V ₂ (PO ₄) ₃	1 M NaPF ₆ /diglyme					
Cu₂NiZn@CNT	22 (2 mA cm ⁻²)/	Na/Cu ₂ NiZn@CN1	[] 20 μL mA h ⁻¹	AlO _x -PE	1	0.2	351.6/1335.5	
	0 (1 mA cm ⁻²)	NaVPO₄F	1 M NaPF ₆ /diglyme					This
FeCoNiZn@CNT	40 (2 mA cm ⁻²)/							work
FeCoNiAlZn@CNT	30 (2 mA cm ⁻²)/							

 Table S3. The comparison of the cell behaviors reported in previous studies.

Materials	Chemical	Purity, %	Manufacturer	Price
	formula			(reagent/industrial grade) (\$
				kg ⁻¹)
Copper nitrate	Cu(NO ₃) ₂ ·3H ₂ O	99.0%-102.0%	Sinopharm Chemical	22.2/4.5
trihydrate			Reagent Co., Ltd.	
Nickel nitrate	Ni(NO ₃) ₂ ·6H ₂ O	≥98.0	Sinopharm Chemical	33.6/3.3
hexahydrate			Reagent Co., Ltd.	
Zinc nitrate	Zn(NO₃)₂·6H₂O	99.0%	Sinopharm Chemical	18.1/3.5
hexahydrate			Reagent Co., Ltd.	
Citric acid monohydrate	$C_6H_8O_7 \cdot H_2O$	≥99.5%	Sinopharm Chemical	10.1/1.2
			Reagent Co., Ltd.	
Ammonia water	NH ₄ OH	25%-28%	Sinopharm Chemical	7.7/0.3
			Reagent Co., Ltd.	
Iron nitrate	$Fe(NO_3)_3 \cdot 9H_2O$	≥98.5%	Sinopharm Chemical	8.3/0.65
nonahydrate			Reagent Co., Ltd.	
Cobalt nitrate	$Co(NO_3)_2 \cdot 6H_2O$	≥98.5%	Sinopharm Chemical	129.2/8.5
hexahydrate			Reagent Co., Ltd.	
Aluminum nitrate	Al(NO ₃) ₃ ·9H ₂ O	≥99.0%	Sinopharm Chemical	26.7/2
nonahydrate			Reagent Co., Ltd.	
Acetylene	C_2H_2		Shaanxi qintang new	2.5 L ⁻¹
			era gas Co., Ltd.	

Table S4. Prices of the main chemical reagents required for the preparation of the substrate

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