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- 1 Supplementary Information for
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- 3 Thermodynamically Driven Self-formation of Ag Nanoparticles in Zn-
- 4 embedded Carbon Nanofibers for Efficient Electrochemical CO₂ Reduction
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- 20~ Fig. S1. Temperature dependence of the composition and structure of the catalyst. FESEM image of AgZn-
- 21~ CNF annealed at (a), (b) 700 °C, (d), (e) 800 °C and (g), (h) 900 °C and TEM image of AgZn-CNF annealed at
- 22 (c) 700 °C, (f) 800 °C and (i) 900 °C.
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 $\,$ Fig. S2. Temperature dependence of the composition of the catalyst. Cross-sectional FESEM image and

26 EDS mapping of AgZn-CNF annealed at (a), (b) 700 °C, (c), (d) 800 °C and (e), (f) 900 °C. Pointed white dots

 $27 ~~{\rm of\ cross-\ sectional\ image\ represent\ Ag\ nanoparticles.}$





Fig. S3. Spectrum and concentration of EDS mapping annealed at (a) 700 °C, (b) 800 °C and (c) 900 °C.



- 33 Fig. S4. (a) cs-STEM image of Zn-CNF with white dots representing atomically dispersed Zn, (b) HAADF-
- $\,$ STEM image of Zn-CNF and EDS analysis of (c) C (d) Zn. $\,$



Fig. S5. (a) first derivative of XANES data to investigate the oxidation states of Zn according to the

 $\,$ materials, (b) EXAFS in k spaces of Zn K-edge.



41~ Fig. S6. XPS analysis (a) deconvolution result of N 1s of w/o metal, Ag-CNF and Zn-CNF (b) deconvolution

- 42~ result of N 1s of w/o metal, Ag-CNF and Zn-CNF.



Fig. S7. XPS deconvolution analysis of AgZn-CNF (a) C 1s (b) O 1s (c) N 1s (d) Ag 3d (e) Zn 2p.





Fig. S8. Faradaic efficiency of electrocatalytic CO_2 reduction of bare CNF.



AgZn-CNF	C (at%)	Ag (at%)	Zn (at%)	O (at%)	Total (at%)
700 °C	85.61	1.12	2.68	10.59	100
800 °C	94.38	0.71	2.63	2.28	100
900 °C	97.77	0	0.28	1.95	100

Table S1. Temperature dependence of atomic composition of AgZn-CNF based on FESEM EDS.

Sample	C (at%)	N (at%)	Ag (at%)	Zn (at%)	O (at%)	Total(at%)
Ag-CNF	87.1	9.7	0.7	0	2.6	100.1
Zn-CNF	86.4	9.3	0	0.7	3.5	99.9
AgZn-CNF	86.9	9.3	0.4	0.9	2.6	100.1

59 Ta	ole S2. XPS	analysis	for ver	ification	of chemica	I composition.
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