Supporting Information

Thermal protocol-driven oxidative etching of ZrC embedded carbon nanofibers to unlock ZrOxCy nanodomains for superior H2O2 production

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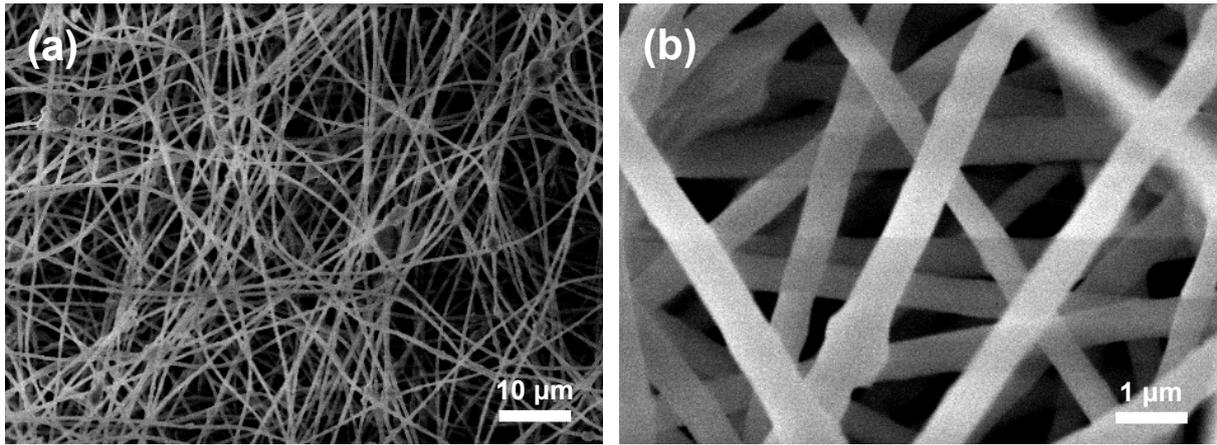
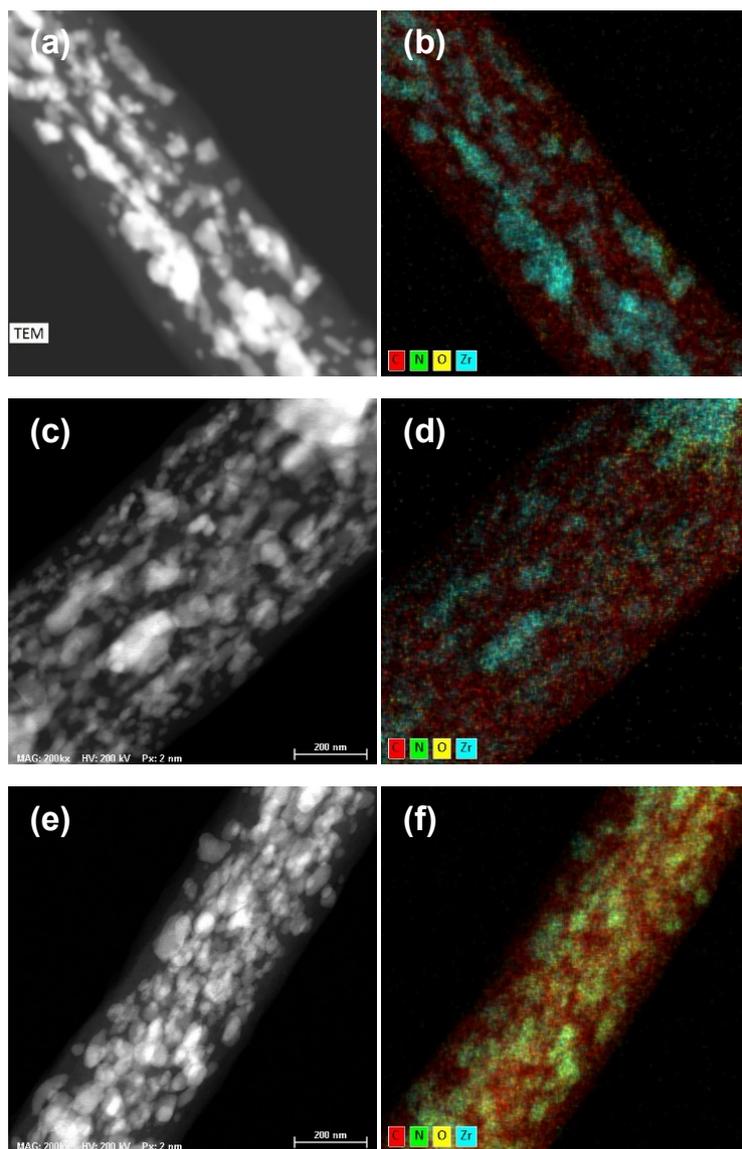**Figure S1.** SEM images of as-spun nanofibers: (a) low magnified and (b) high magnified images.

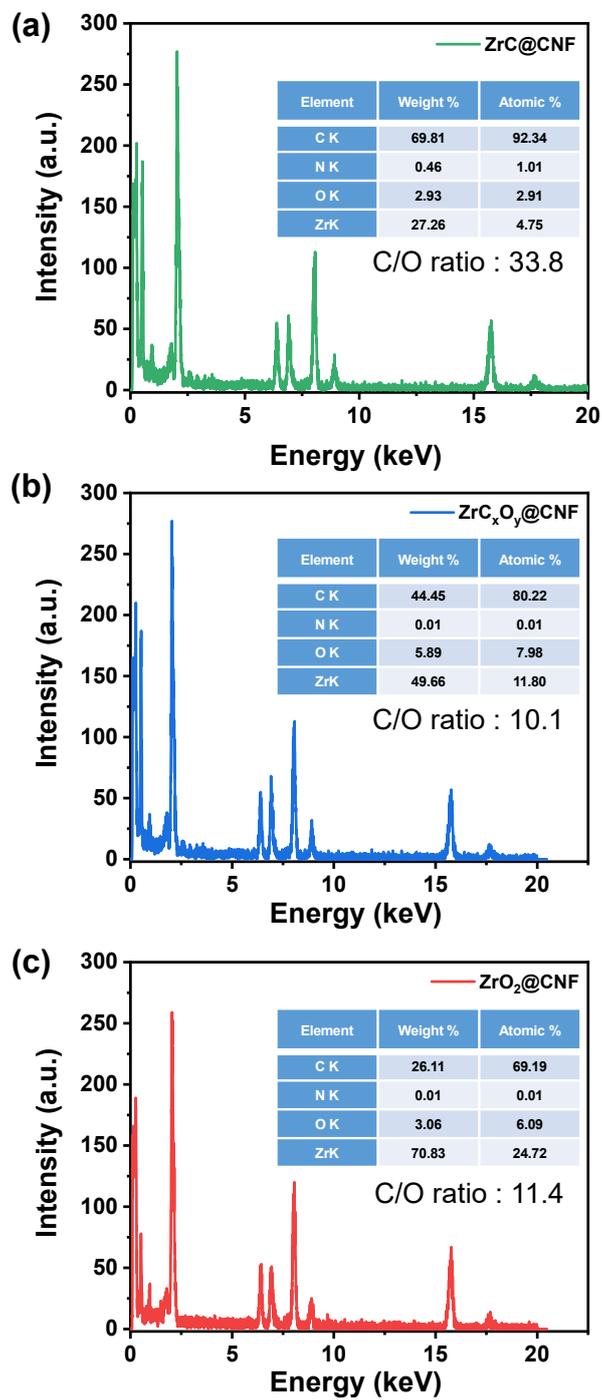**Figure S2.** STEM-EDS mapping analysis of (a,b) ZrC@CNF, (c,d) ZrC$_x$O$_y$@CNF, and (e,f) ZrO$_2$@CNF.

(a)

Element	Weight %	Atomic %
C K	69.81	92.34
N K	0.46	1.01
O K	2.93	2.91
ZrK	27.26	4.75

C/O ratio : 33.8

ZrC@CNF

(b)

Element	Weight %	Atomic %
C K	44.45	80.22
N K	0.01	0.01
O K	5.89	7.98
ZrK	49.66	11.80

C/O ratio : 10.1

ZrC$_x$O$_y$@CNF

(c)

Element	Weight %	Atomic %
C K	26.11	69.19
N K	0.01	0.01
O K	3.06	6.09
ZrK	70.83	24.72

C/O ratio : 11.4

ZrO$_2$@CNF

Figure S3. EDS results of (a) ZrC@CNF, (b) ZrO$_x$C$_y$@CNF, and (c) ZrO$_2$@CNF catalysts.
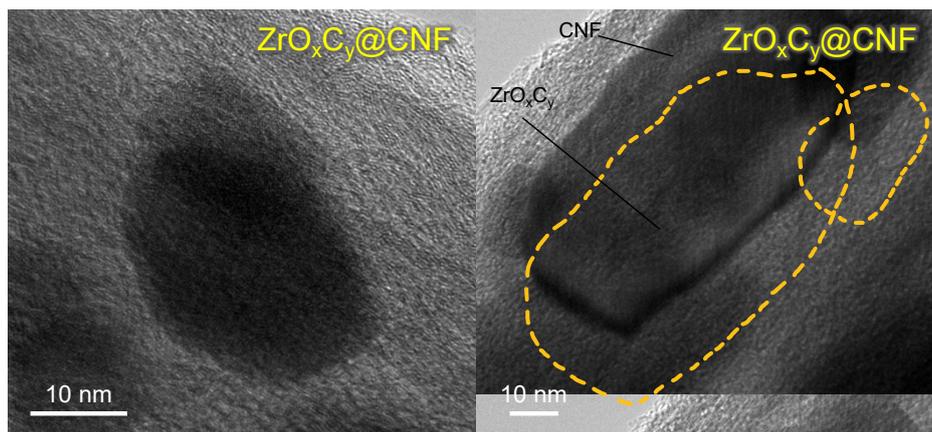
Figure S4. High-magnification TEM images of Zr-containing nanodomains embedded in the CNF matrix.

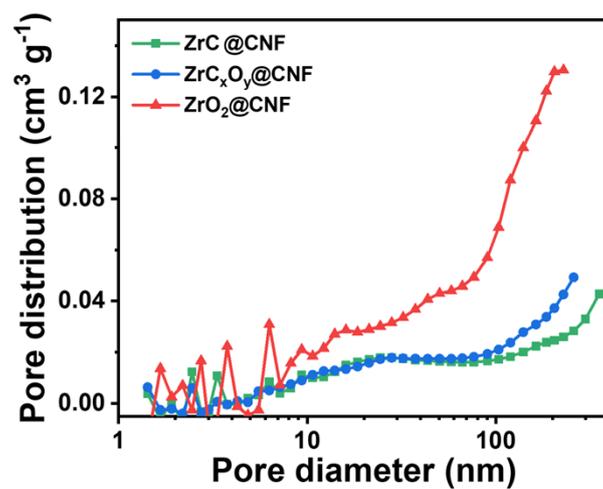**Figure S5.** (a) TGA and DSC graphs of ZrC@CNF, ZrO_xC_y@CNF, and ZrO_2@CNF.

Figure S6. BJH plots of ZrC@CNF, ZrO$_x$C$_y$@CNF, and ZrO$_2$@CNF for pore size distribution.
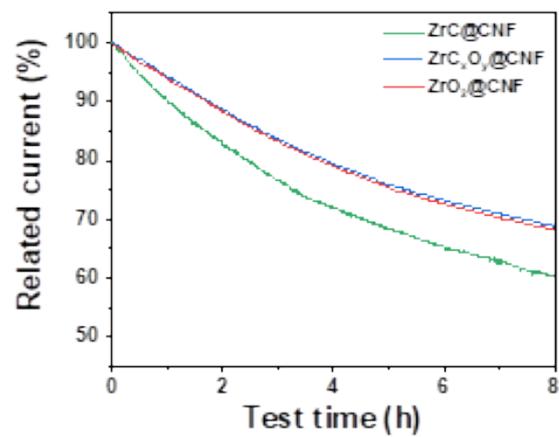
Figure S7. Chronoamperometry test of ZrC@CNF, ZrC$_x$O$_y$@CNF, and ZrO$_2$@CNF.

Table S1. Comparison of the electrochemical performance between the ZrO_2@CNF and the recently reported electrocatalysts.

Samples	Electrolytes	H_2O_2 selectivity (%)	Mass activity (A g^{-1})	Electro transfer number	Onset potential (V vs. RHE)	Ref
Au-Pt-Ni	0.1M KOH	95	192.9	2.11	0.73	1
Fe-C-O	0.1M KOH	95	N/A	2.09	0.822	2
$Ba_{0.5}Sr_{0.5}Fe_{(1-x)}Cu_xO_{3-\delta}$	0.1M KOH	65	N/A	3.1	0.7	3
BCN	0.1M KOH	70	N/A	2.5	0.83	4
O-CNTs	0.1M KOH	90	N/A	N/A	0.75	5
Co-NG(O)	0.1M KOH	82	284±22	N/A	0.85	6
Co-Nx-C	0.1M KOH	80	N/A	N/A	0.84	7
ZrO_2@CNF	0.1M KOH	85.3	14.0	2.3	0.75	This work

Table S2. Atomic percentage (at%) values obtained from XPS peak deconvolution.

	ZrC@CNF (At %)	ZrCₓOᵧ@CNF (At %)	ZrO₂@CNF (At %)
Zr 3d			
Zr-O	52.48	87.97	100
Zr-C	47.52	12.03	-
O 1s			
-OH	24.24	10.74	5.50
O_V	27.87	39.31	41.11
O_L	47.89	49.95	53.39
C 1s			
sp^2	76.22	27.78	37.21
sp^3	4.62	29.61	28.49
C-O	10.04	33.94	25.64
O-C=O	9.12	8.67	8.66

Supporting reference

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