Supporting Information

Ni clusters immobilized on oxygen-rich siloxene nanosheets for efficient electrocatalytic oxygen reduction toward H_2O_2 synthesis

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Supporting Figures

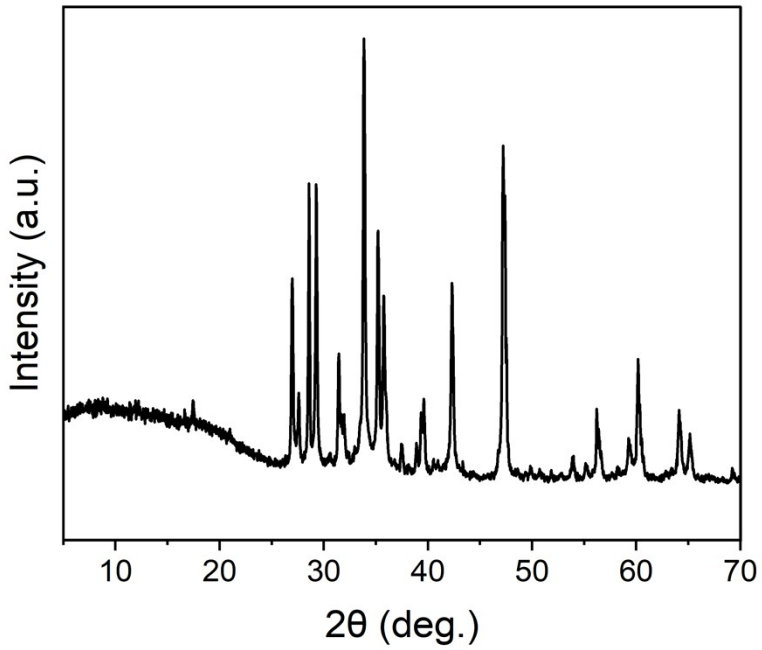

Figure S1. PXRD pattern of $CaSi_2$.

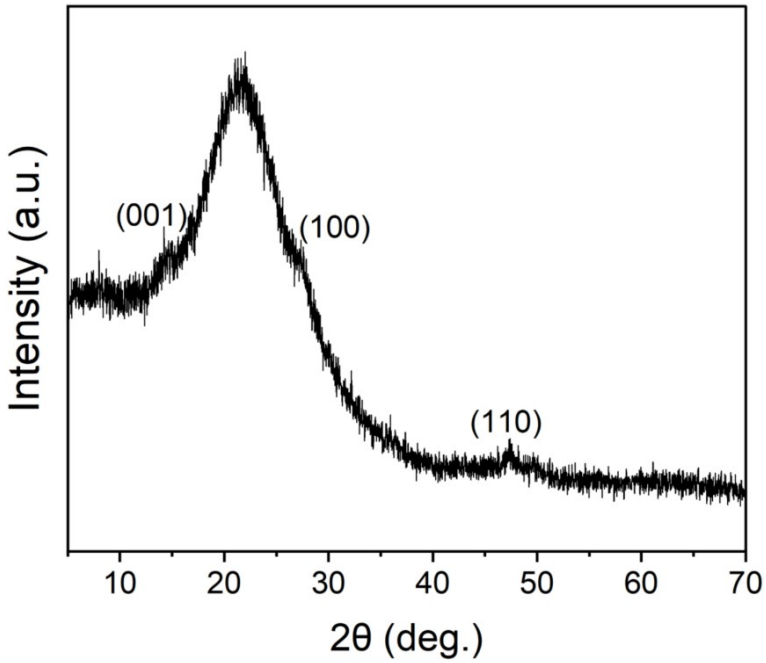

Figure S2. PXRD pattern of siloxene.

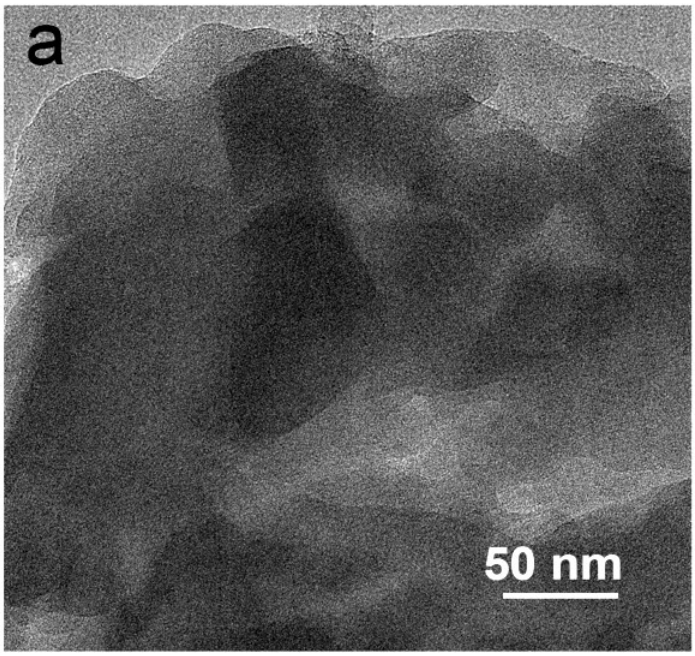

Figure S3. TEM image of siloxene.

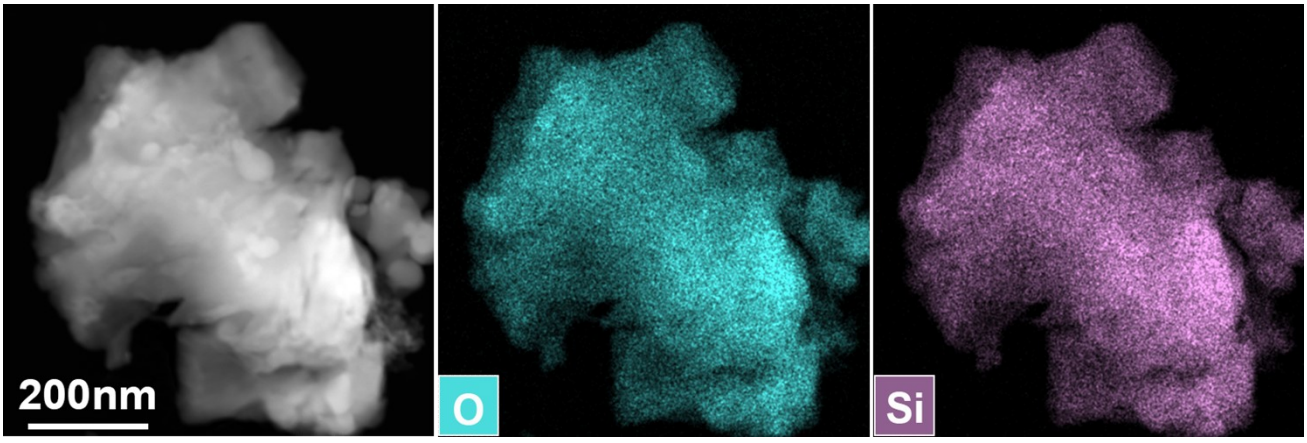

Figure S4. EDS elemental mappings of siloxene.

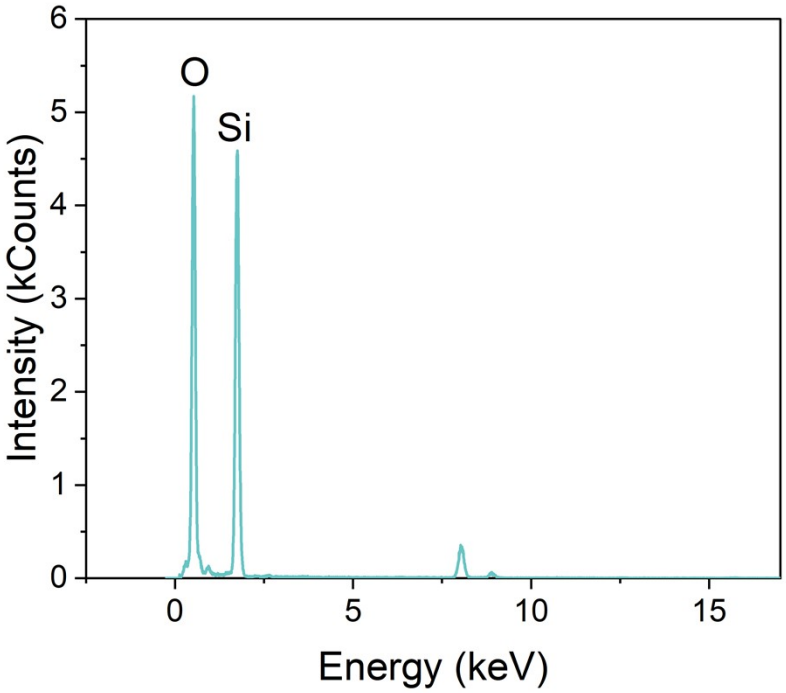

Figure S5. EDS spectrum of Ni/siloxene.

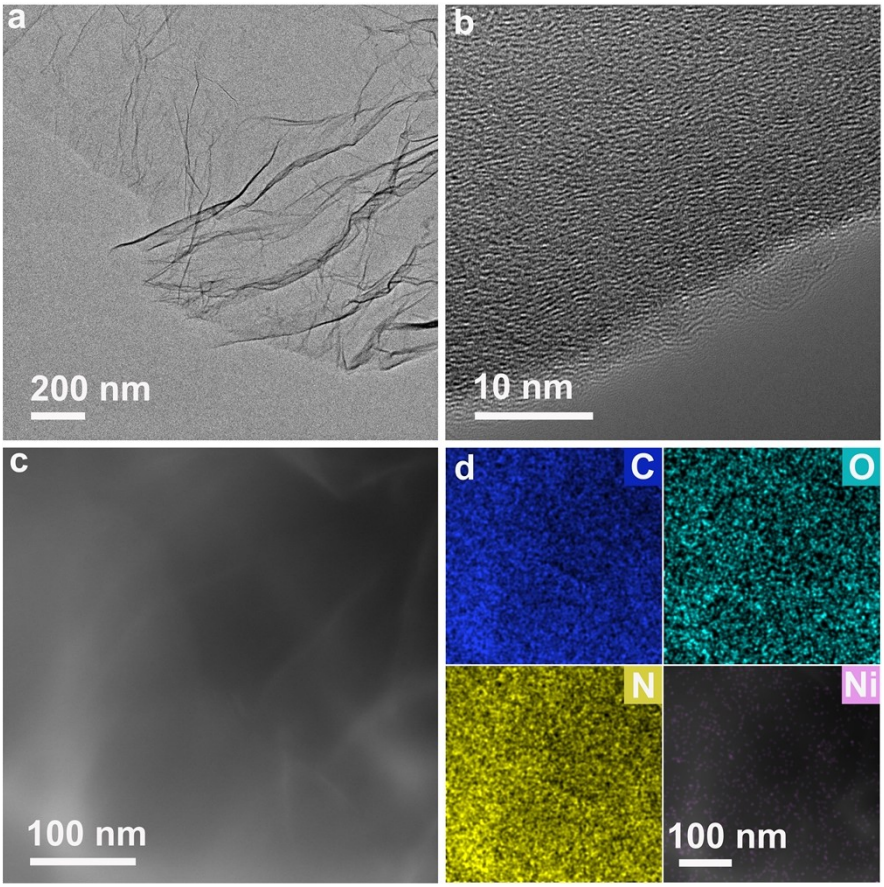

Figure S6. TEM images and EDS elemental mappings of Ni_{SA}/rGO. (a, b) TEM images at different magnifications. (c) HAADF image and (d) mappings of C, O, N and Ni elements.

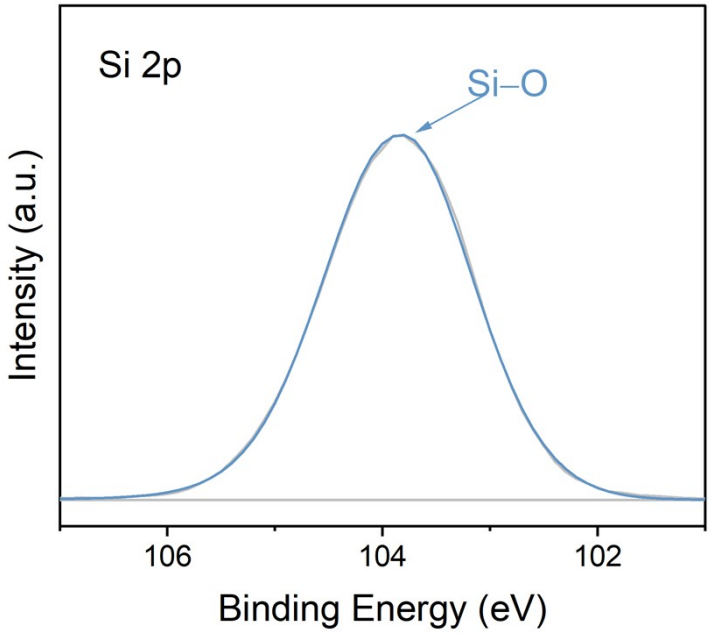

Figure S7. High-resolution Si 2p XPS spectrum of Ni/siloxene.

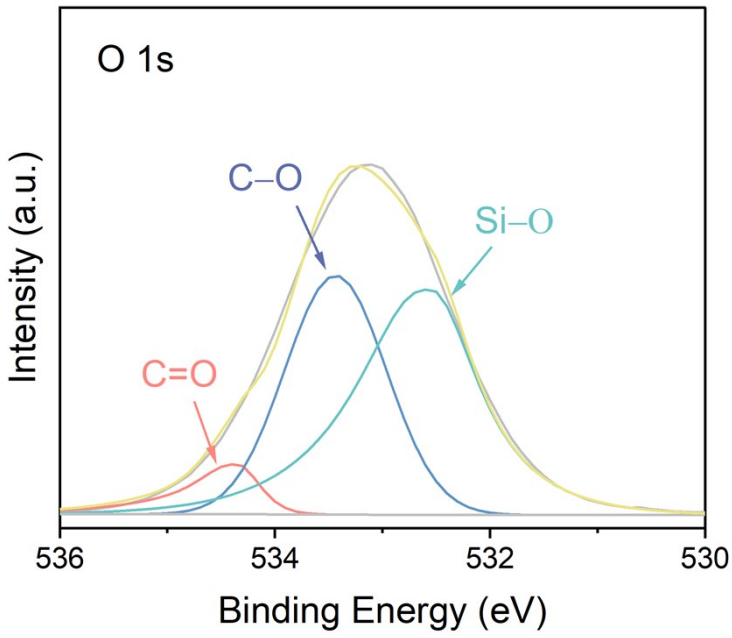

Figure S8. High-resolution O 1s XPS spectrum of Ni/siloxene.

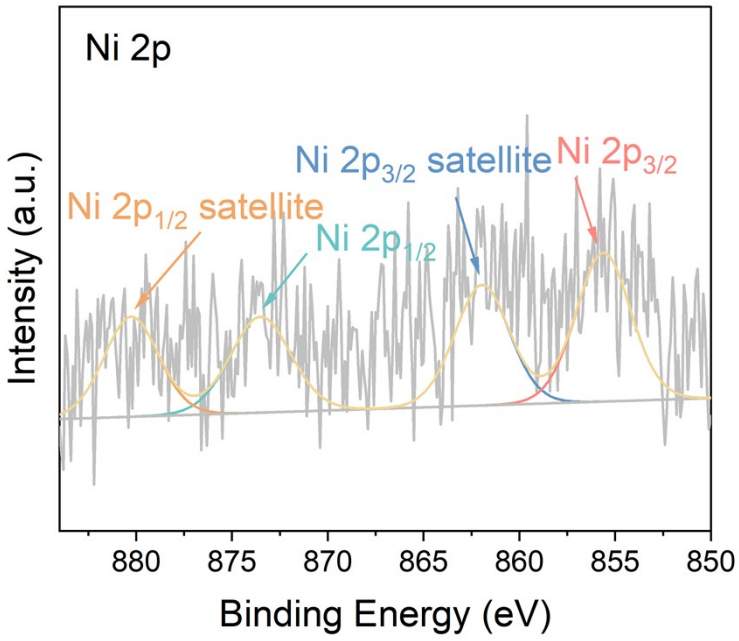

Figure S9. High-resolution Ni 2p XPS spectrum of Ni/siloxene.

Supporting Tables

Table S1. EXAFS fitting parameters at the Ni *K*-edge for Ni/siloxene (S_0^2 = 0.72) and Ni_{SA}/rGO (S_0^2 = 0.80).

Sample	Path	CN	R (Å)	σ^2 (Å^2)	ΔE_0 (eV)	R factor
Ni/siloxene	Ni–Ni	4.5	2.493±0.019	0.002±0.001	7.016±3.028	0.013
Ni_{SA}/rGO	Ni–N	2.0±0.29	1.855±0.015	0.007±0.002	4.824±1.917	0.014
	Ni–Ni	1.0	2.505±0.023	0.010±0.003	4.824±1.917	0.014

CN: coordination numbers; R: bond distance; σ^2: Debye-Waller factors; ΔE_0: the inner potential correction; R factor: goodness of fit

Table S2. Comparison of alkaline 2e⁻ ORR electrocatalysis for H_2O_2 synthesis of Ni/siloxene with other electrocatalysts reported.

Sample	H_2O_2 Selectivity	E_{onset} (V)	i_{ring}(mA)@0.2 V	j_{disk} (mA cm^{-2})@0.2 V	Reference
Ni/siloxene	**100%@0.3-0.65 V**	**0.75**	**0.12**	**–1.47**	**This work**
Ni-N-C	52%	0.72	0.07	–2.00	*J. Am. Chem. Soc.*, 2019[1]
N-CBMC-500	92%-95.9%@0-0.6 V	0.80	0.30	–2.00	*J. Mater. Chem. A*, 2022[2]
CMK-3	80%-55%@0.1-0.4 V	0.40	0.02	–0.50	*ACS Catal.*, 2018[3]
Ni_4-B_1@BNC	86%-90%@0.1-0.6 V	0.65	0.16	–2.50	*Small*, 2022[4]
O-BC-2-650	82%-94%@0-0.6 V	0.78	—	–3.00	*Sci. China Mater.*, 2022[5]
Co-N_5 SACs	80%-85%@0.3-0.75 V	0.82	0.35	–2.90	*Appl. Catal. B: Environ.*, 2023[6]
OXO-G/NH_3·H_2O	82%-78%@0.1-0.7 V	0.80	0.20	–3.10	*ACS Catal.*, 2019[7]
APES-Au/PC	95%-100%@0.1-0.5 V	0.80	0.20	–2.42	*Chem. Eng. J.*, 2022[8]
CoNOC	70%-100%@0.05-0.6 V	0.60	0.40	–3.10	*Angew. Chem. Int. Ed.*, 2023[9]
Co-N_2-C/LO	80%-90%@0-0.75 V	0.80	0.39	–2.70	*Adv. Funct. Mater.*, 2022[10]
HCNFs	88%-97%@0.2-0.7 V	0.85	0.22	–3.20	*Angew. Chem. Int. Ed.*, 2021[11]
Nb_2CT_x	86%-90%@0.2-0.6 V	0.75	0.36	–2.18	*Nano Res.*, 2022[12]
Mo-F-C	78%-80%@0.2-0.7V	0.78	0.12	–2.80	*J. Energy Chem.*, 2023[13]
Co-F-CNT	72%-90%@0.1-0.8 V	0.75	0.15	–3.80	*EcoMat.*, 2023[14]

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