Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C.

Supporting information for

Three-Dimensional Nano-folded Transition-Metal Oxide Electrode Materials for High-Performing Electrochemical Energy Storage Devices

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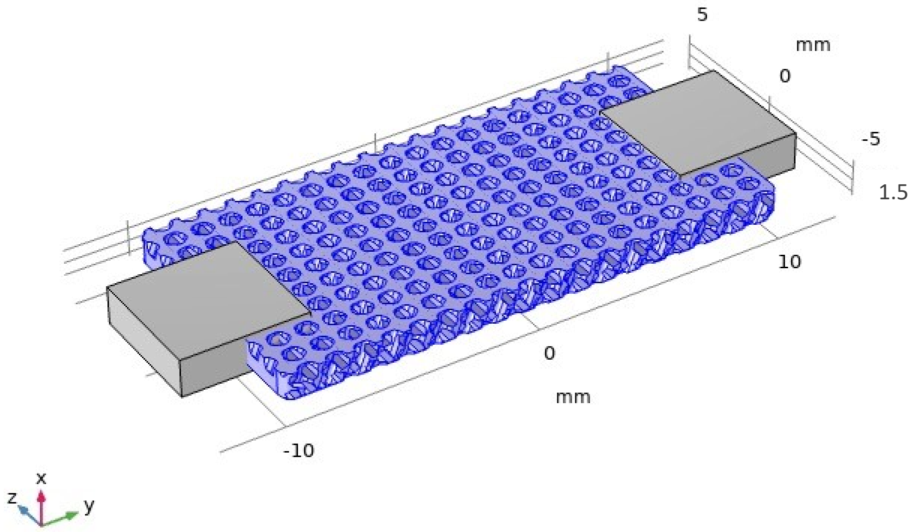

Figure S1. The model and geometry parameters for the dynamic thermal oxidation.

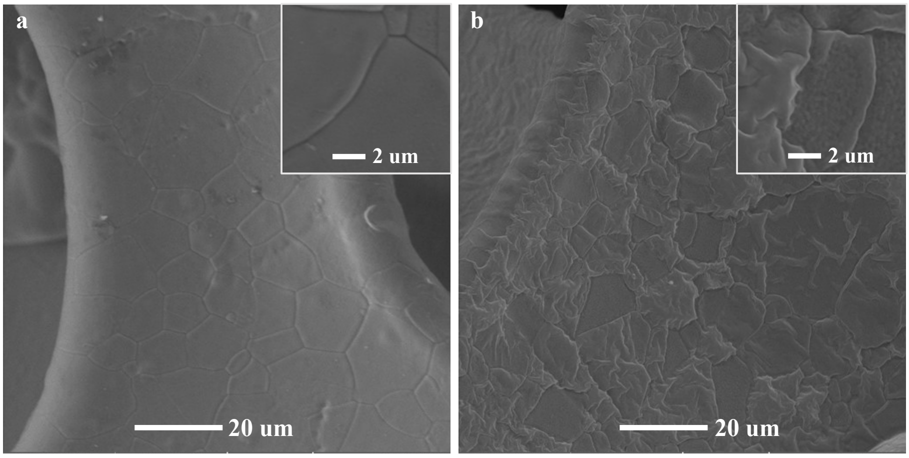

Figure S2. The morphological evolutions of the Ni foam before and after dynamic thermal oxidation. (a) representative SEM images of Ni foam after metallographic corrosion; (b) the view of Ni foam after dynamic thermal oxidation.

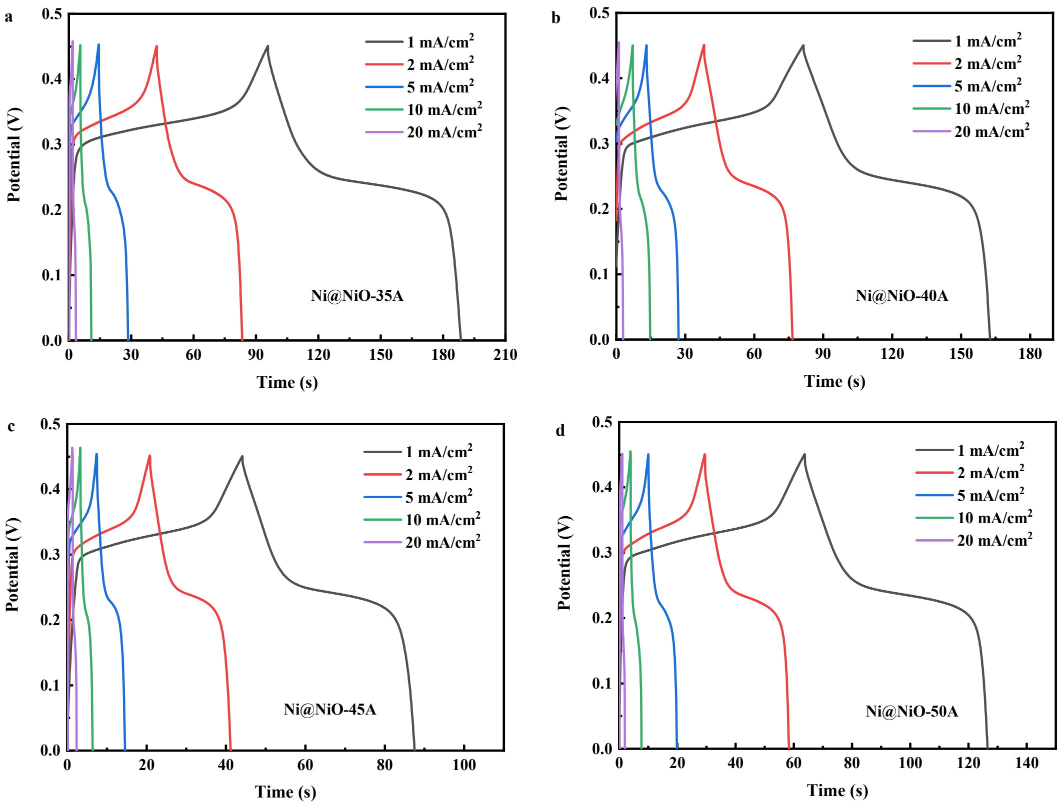

Figure S3. GCD curves of 3D nano-folded Ni@NiO freestanding electrode obtained at different dynamic thermal oxidation stages: (a) Ni@NiO-35A; (b) Ni@NiO-40A; (c) Ni@NiO-45A; (d) Ni@NiO-50A).

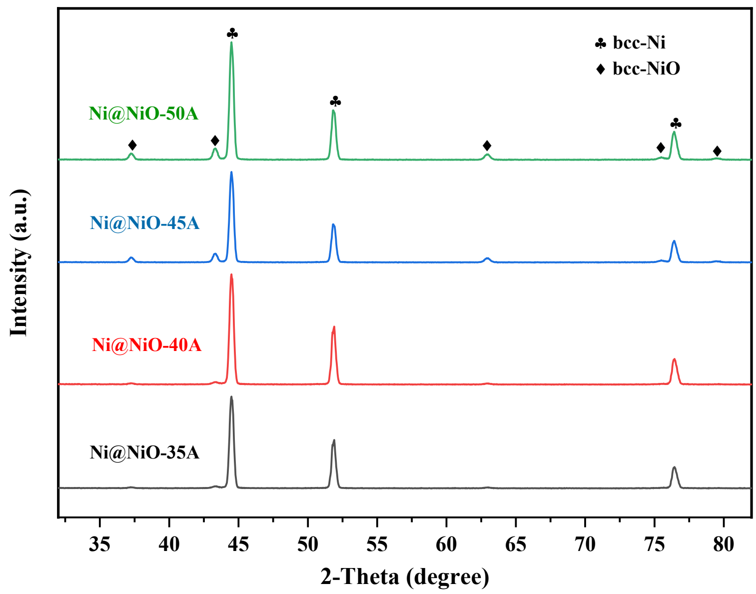

Figure S4. XRD pattern of the 3D nano-folded Ni@NiO obtained at different conditions.

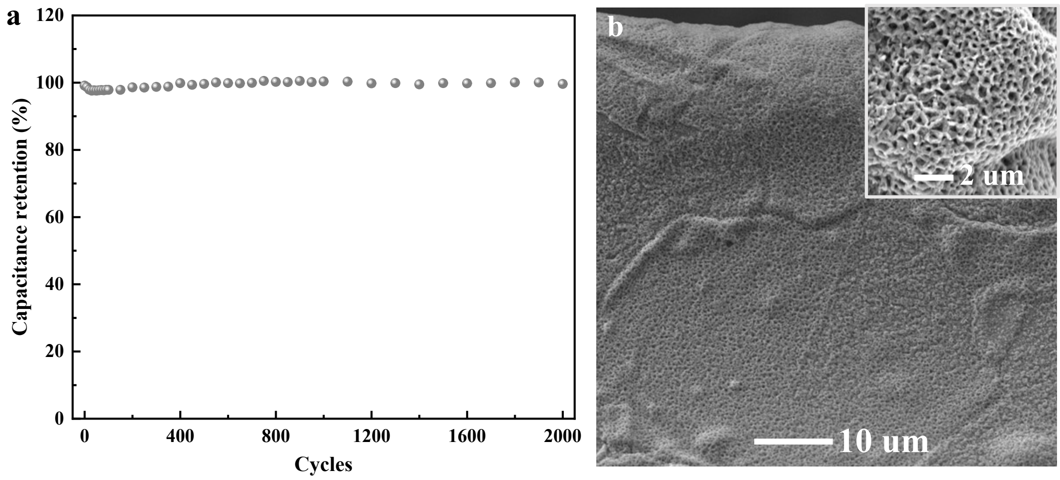

Figure S5. The cycling performance and morphological evolution of the Ni@NiO-40A electrode. (a) Capacitance retention with the cycling operations; (b) representative SEM view of the Ni@NiO-40A electrode after 2000 cycles.

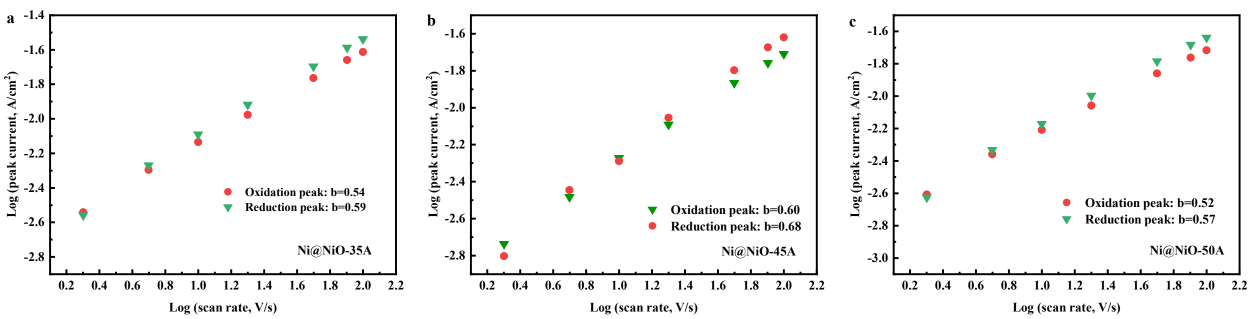

Figure S6. The oxidation and reduction peak derived *b*-value at different scan rates of the 3D nano-folded Ni@NiO freestanding electrode obtained at different dynamic thermal oxidation stages.

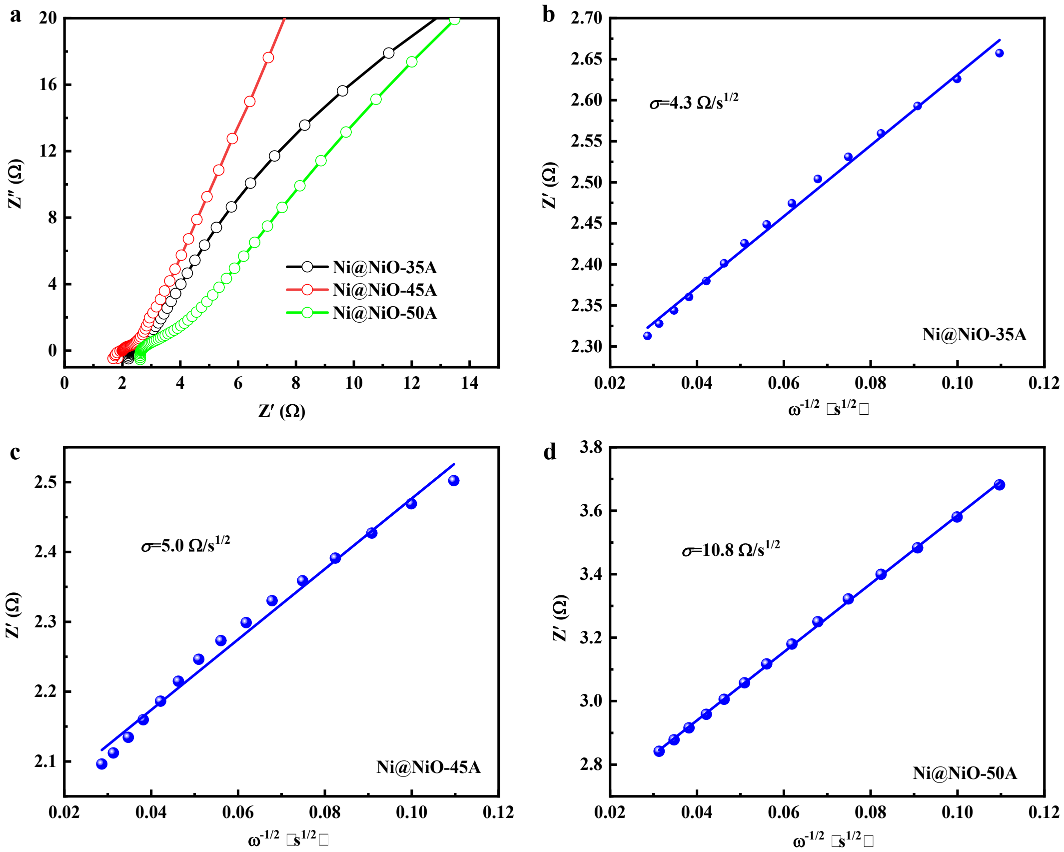

Figure S7. Electrochemical kinetics of the 3D nano-folded Ni@NiO freestanding electrode obtained at different dynamic thermal oxidation stages. (a) EIS spectrum of the different samples; (b-c) The relationship between *Z′* and the reciprocal of the square root of frequency ($\omega^{-1/2}$) in the intermediate frequency range.

Table S1. NiO coverage and stoichiometric analysis in the 3D nano-folded Ni@NiO obtained at different condition.

Samples	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Spot 6	Average
Ni@NiO-35A	1.15	1.02	0.98	1.09	1.03	1.21	1.08
Ni@NiO-40A	1.04	0.96	1.01	1.05	0.98	1.03	1.01
Ni@NiO-45A	0.94	0.92	1.06	0.97	0.89	1.01	0.97
Ni@NiO-50A	0.98	0.86	0.92	1.03	0.96	0.94	0.95

Table S2. Electrochemical performance of recent reported NiO-based electrode materials.

Electrodes	Capacitance	Rate performance	Ref.
3D nano-folded Ni@NiO	0.23 F/cm^2 at 2 mV/s (~295 F/g)	50% (from 2 mV/s to 50 mV/s)	Our work
NiO-carbon cloth	132 F/g at 5 mV/s	32% (from 5 mV/s to 50 mV/s)	[1]
NiO Fine Nanoparticles	243 F/g at 2 mV/s	33% (from 2 mV/s to 50 mV/s)	[2]
NiO Nanoflakes	263 F/g at 5 mV/s	48% (from 2 mV/s to 50 mV/s)	[3]

The mass loading of the electrochemical active NiO in the 3D nano-folded Ni@NiO can be calculated by weighting the mass changes of Ni nanofoams before and after oxidation, which was operated by a microbalance (Mettler, XS105DU) with an accuracy of 0.01 mg. Due to the oxidation formula 2Ni + O_2 —> 2NiO, the weights of NiO (m_{NiO}) in the final product 3D nano-folded Ni@NiO can be derived from $m_{NiO} = \Delta m * 74.69/16$ (Δm denotes the weight difference of Ni nanofoams before and after oxidation). Accordingly, the mass loading of electrochemical active NiO in the 3D nano-folded Ni@NiO obtained at different condition was calculated as listed in the Table S1.

Table S3. NiO contents in the 3D nano-folded Ni@NiO obtained at different condition.

	Ni@NiO-35A	Ni@NiO-40A	Ni@NiO-45A	Ni@NiO-50A
m_{NiO} (mg)	0.47	0.78	0.97	1.35
NiO Content percentage	7.8%	13%	16.2%	22.5%

Reference

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