

Supporting Information

Synthesis of Hierarchically Porous TiNb_2O_7 Nanotubes with Controllable Porosity and Their Application in High Power Li-Ion Batteries

Hyunjung Park¹, DongHyeok Shin¹, Taeseup Song^{2}, Won Il Park^{3*}, and Ungyu Paik^{1*}*

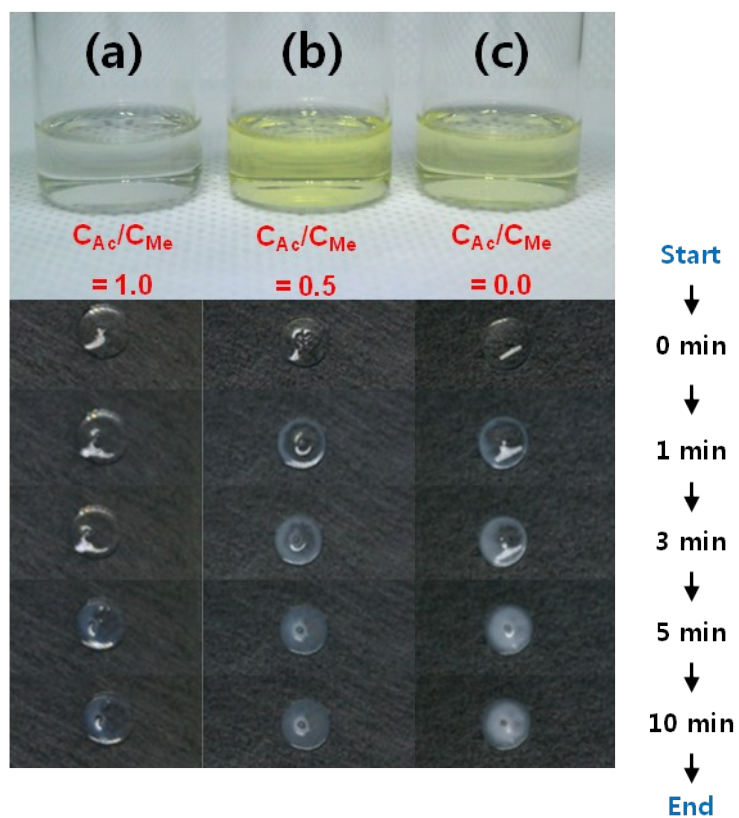


Fig. S1 Observation of a hydrolysis degree on three types of mixture solutions prepared with different molar ratios of acetic acid to Ti(IV)/Nb(V) precursors in ethanol. (a) $C_{Ac}/C_{Me} = 0$, (b) $C_{Ac}/C_{Me} \approx 0.5$, and (c) $C_{Ac}/C_{Me} \approx 1$; the molar concentrations of metal precursors in the solutions are fixed at 0.5 M.

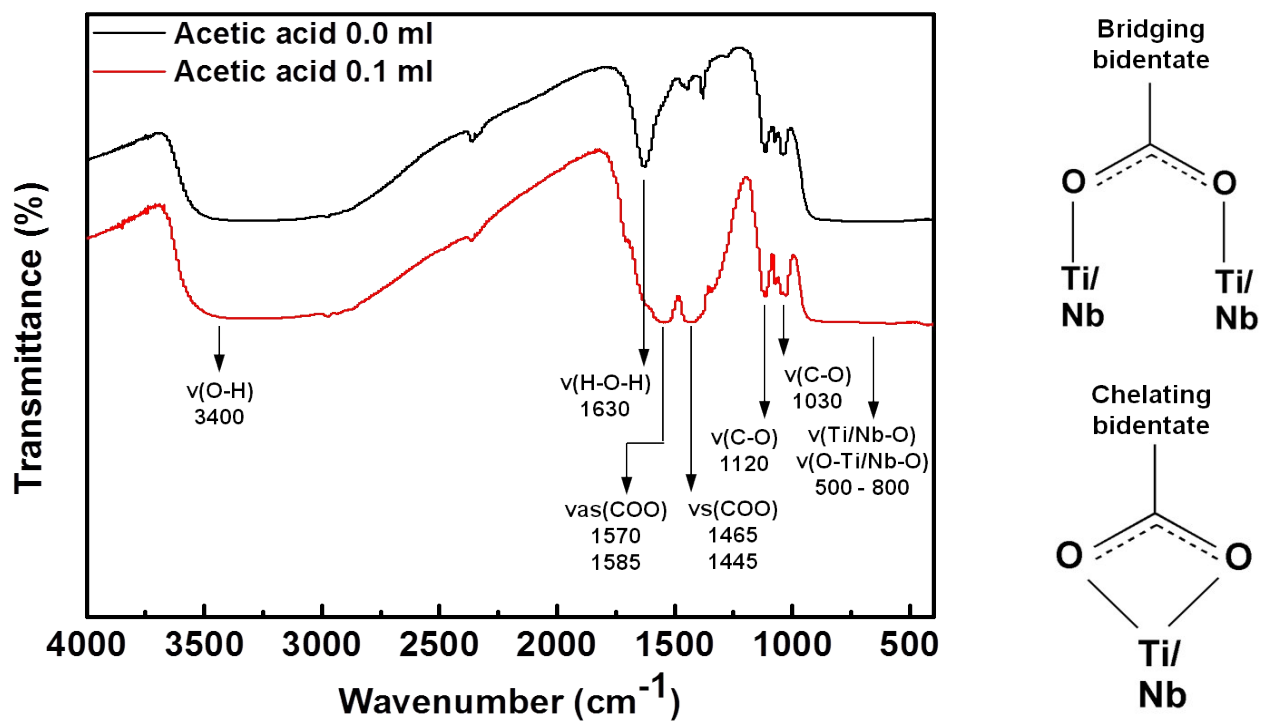


Fig. S2 FTIR spectra of powders prepared by Ti (IV)/Nb (V) alkoxide precursors and acetic acid after drying at 100 °C in air.

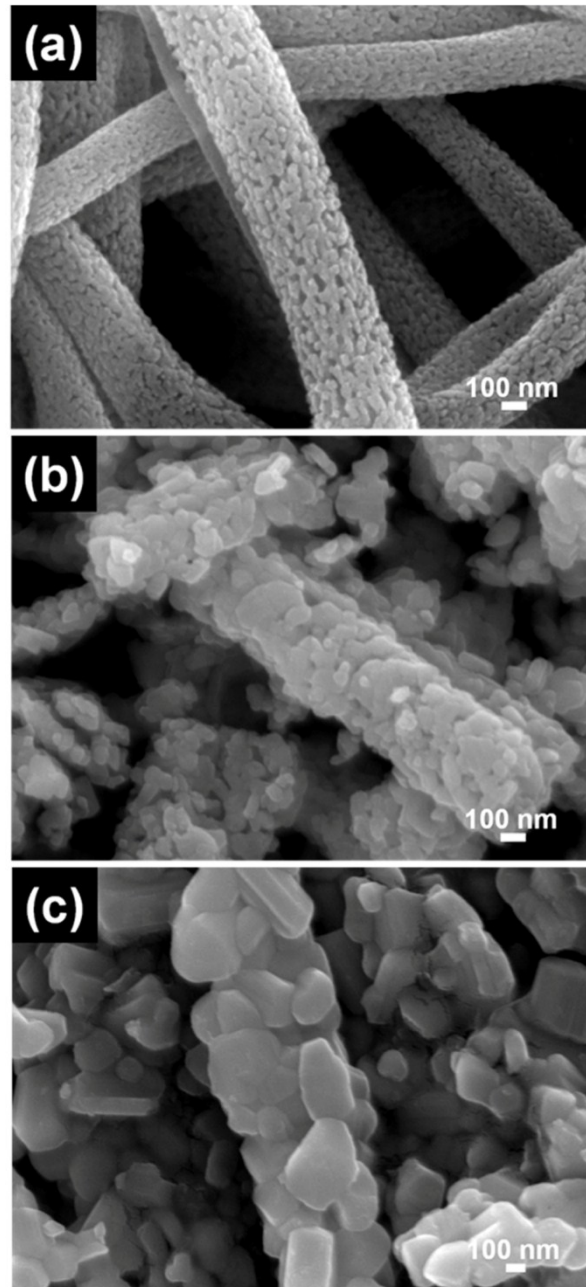


Fig. S3 SEM images of TNO NTs-2 calcined at different temperatures: (a) 700 °C, (b) 850 °C, and (c) 1000 °C.

Sample	2theta	FWHM	β	Crystallite size ; D (nm)	mean (nm)
TNO NTs-1	39.10	1.08	0.019	7.7	7.8
	47.65	1.75	0.019	7.9	
TNO NTs-2	39.00	0.55	0.010	16.3	16.4
	47.70	0.52	0.009	16.5	
TNO NTs-3	39.02	0.59	0.010	14.1	14.9
	47.95	0.55	0.009	15.6	

Table 1. Calculation of mean particle sizes of TNO NTs based on Scherrer equation.

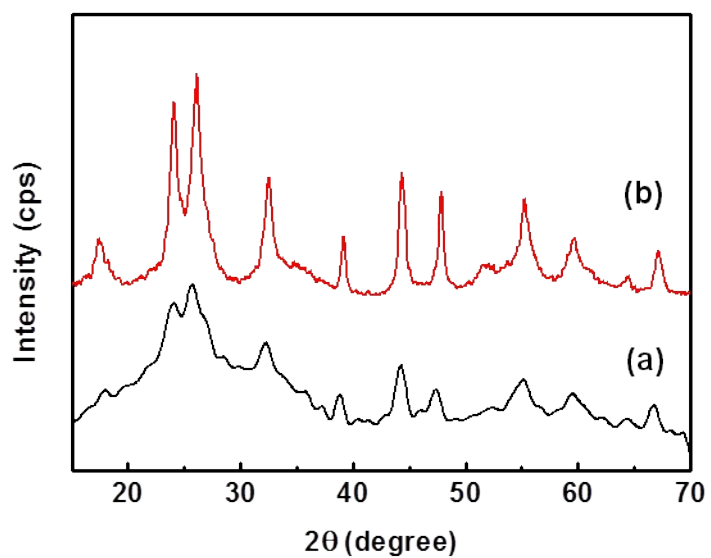


Fig. S4 XRD peak patterns of TNO NTs-1 calcined at different temperatures in air for 2h: (a) 700 °C and (b) 750 °C

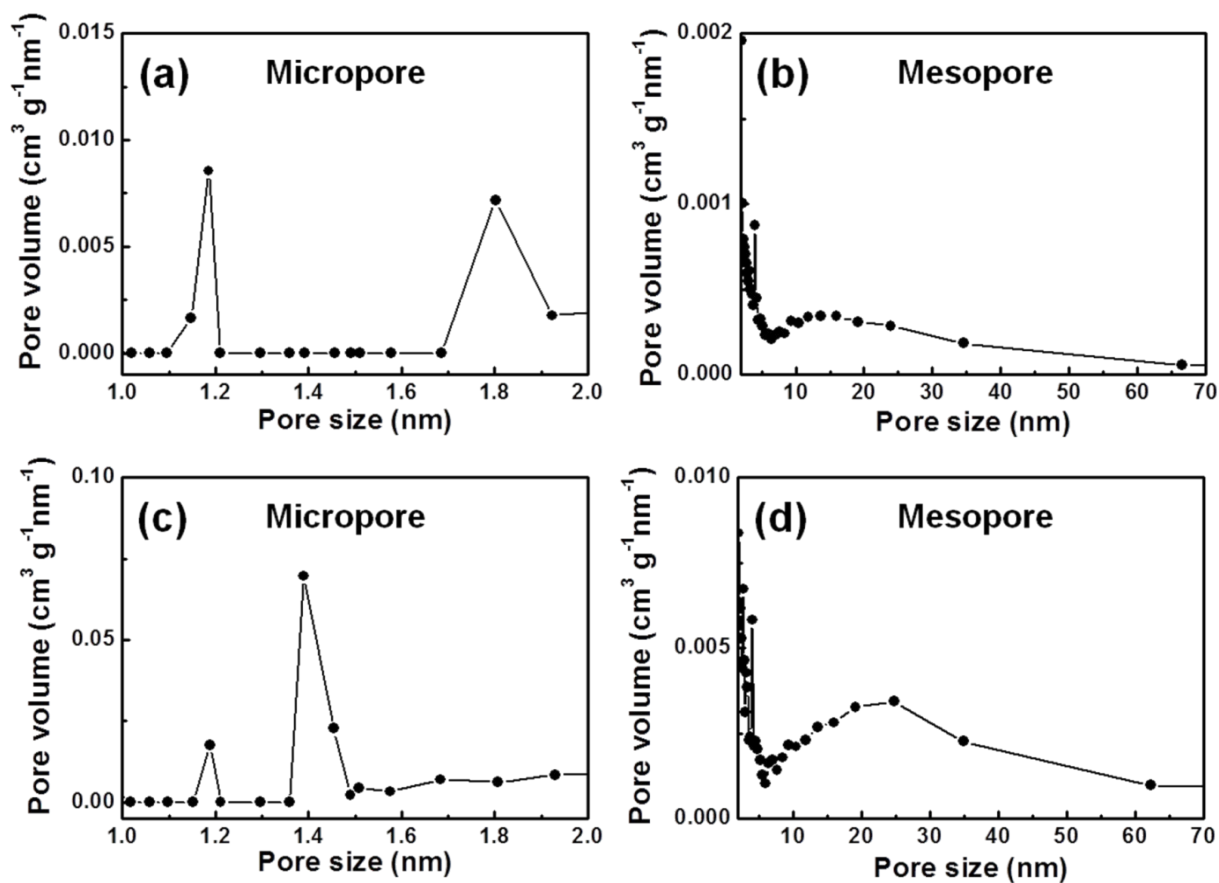


Fig. S5 Pore size distribution in the ranges of micro ($d < 2$ nm) and mesopore ($2 \text{ nm} < d < 50$ nm): (a) and (b) TNO NTs-2, and (c) and (d) TNO NTs-3.

Material	Morphology	Template	Calcination temperature (°C)	Surface area (m ² g ⁻¹)	Reference number
TiNb ₂ O ₇	Particles	F127	700	85	40
TiO ₂	Nanofibers	PVP (M _w ≈ 1,300,000), F127	540	90	50
TiO ₂	Nanofibers	P123	600	32	51
TiO ₂	Nanofibers	PVP (M _w ≈ 1,300,000), ZnO	500	149	52
TiO ₂	Hollow nanofibers	PMMA (M _w ≈ 120,000), CTAB	500	59	53
TiO ₂	Nanofibers	F127	450	112	54
TiO ₂	Nanofibers	PVP (M _w ≈ 1,300,000), Carbon spheres	500	33	55

Table 2. Comparison of titanium based metal oxides on their surface area prepared with different kinds of templates, chemicals, and calcination temperatures.

Compound	Morphology	1st discharge capacity (mAh g ⁻¹)	Rate capability (mAh g ⁻¹)	Capacity after cycles (mAh g ⁻¹)	Cycle number	Remarks	Reference
Carbon/ TiNb ₂ O ₇	Microparticles	285 @ 0.1C	~ 130 @ 2C	-	-	-	19
TiNb ₂ O ₇	Nanoparticles	213 @ 1C	~ 76 @ 10C	175	300 @ 1C	Particle size: ~ 10 nm	21
TiNb ₂ O ₇	Hierarchical microspheres	~ 300 @ 0.1C	~ 100 @ 20C	115	500 @ 10C	Particle size: 2 - 3 μm	22
TiNb ₂ O ₇	Nanofibers	~ 284 @ 0.1C	~ 60 @ 20C	250	50 @ 1C	Diameter: 500 nm, Particle size: 100 nm	23
TiNb ₂ O ₇	Porous nanofibers	~ 284 @ 1C	~ 170 @ 100C	170	500 @ 5C	Diameter: 110 nm, Particle size: 10 nm	25
TiNb ₂ O ₇ / Ti _{1-x} Nb _x N	Hierarchical microspheres	~ 265 @ 0.1C	~ 143 @ 100C	182	1000 @ 5C	Particle size: 1.2 μm	26
TiNb ₂ O ₇	Porous particles	~ 281 @ 0.1C	~ 125 @ 50C	200	1000 @ 5C	Particle size: 20 - 30 nm	27
TiNb ₂ O ₇	Nanotubes	~ 265 @ 0.1C	~ 85 @ 100C	175	700 @ 1C	TNO NTs-1	This work
TiNb ₂ O ₇	Porous nanotubes	~ 281 @ 0.1C	~ 115 @ 100C	188	700 @ 1C	TNO NTs-2	This work
TiNb ₂ O ₇	Hierarchically porous nanotubes	~ 294 @ 0.1C	~ 180 @ 100C	210	700 @ 1C	TNO NTs-3	This work

Table 3. Comparison of the electrochemical performances of TiNb₂O₇ previously reported.

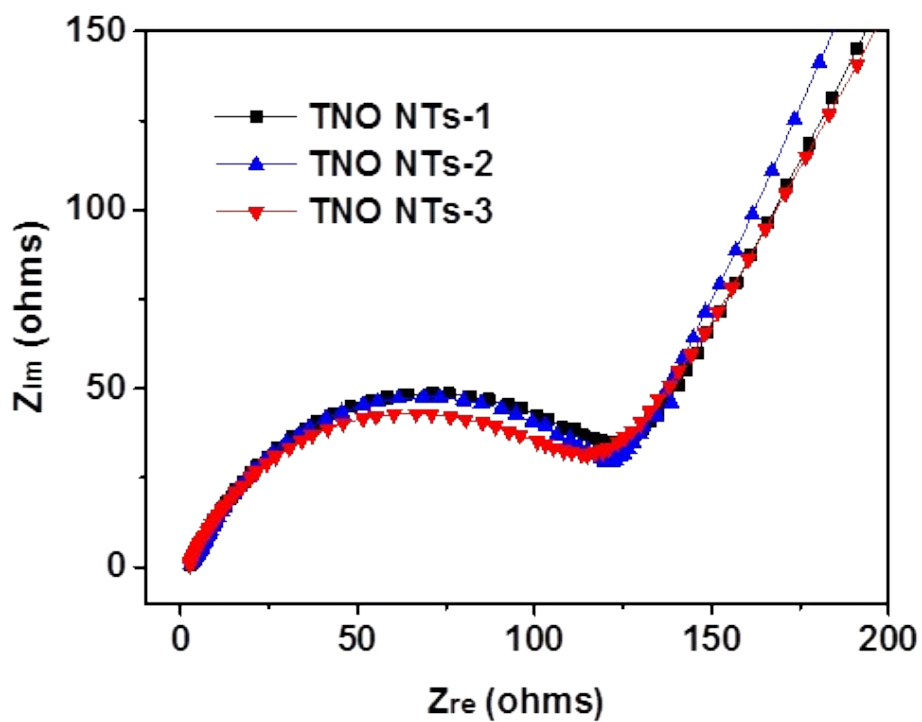


Fig. S6 Electrochemical impedance spectra of TNO NTs-1/2/3 electrodes obtained by sinusoidal wave with an amplitude of 10.0 mV within the frequency range from 200 kHz to 0.1 Hz.