Supporting information

Tailoring the Defects of Sub-100 nm Multipodal Titanium Nitride/Oxynitride Nanotubes for Efficient Water Splitting Performance†

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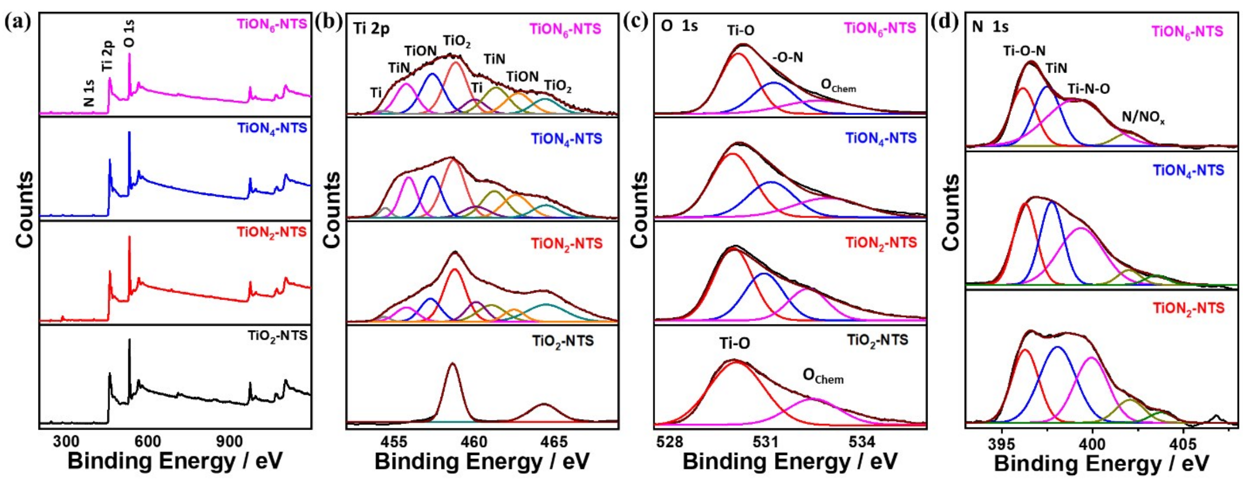

Figure S1. (a) XPS surveys of the as-synthesized materials and their high-resolution XPS spectra of (b) Ti 2p, (c) O 1s, (d) N1s of TiO_2NTs and $TiON_x$NTs after etching.

Table S1. The binding energies of Ti, O, and N as well as the N-content in the as-synthesized materials. The unit for all the values in the table are in (eV)

Samples		Before etching				After etching			
		TiO_2NTs	$TiON_2$NTs	$TiON_4$NTs	$TiON_6$NTs	TiO_2NTs	$TiON_2$NTs	$TiON_4$NTs	$TiON_6$NTs
Ti	Ti	----	----	----	----	----	454.4	454.4	454.4
	Ti	----	----	----	----	----	460.0	460.0	460.0
	TiN	----	455.5	455.6	455.8	----	455.7	455.7	455.7
	TiN	----	461.3	461.4	461.5	----	461.0	461.2	461.3
	Ti-O-N	----	457.2	457.0	456.9	----	457.2	457.3	457.4
	Ti-O-N	----	462.7	462.6	462.5	----	462.5	462.5	462.7
	Ti-O	458.7	458.4	458.4	458.4	458.7	458.7	458.7	458.8
	Ti-O	464.4	464.3	464.3	464.3	464.3	464.4	464.4	464.5
O	Ti-O	530.2	530.5	530.6	530.9	530.2	530.0	530.0	530.1
	O-H	532.1	----	----	----	----	----	----	----
	O-N	----	531.5	531.6	531.7	----	530.9	531.2	531.3
	O_{ads}	532.5	532.7	532.7	532.8	532.5	532.4	533.0	533.0
N	Ti-O-N	----	396.0	395.7	----	----	396.3	396.3	396.1
	TiN	----	397.4	397.8	397.8	----	398.0	397.6	397.5
	Ti-N-O	----	399.0	399.8	399.1	----	399.9	399.3	399.1
	NO	----	401.5	401.3	400.5	----	402.1	402.4	401.9
	NOx	----	402.4	402.7	402.9	----	403.9	----	----
	NOx	----	404.7	404.8	405.1	----	----	----	----

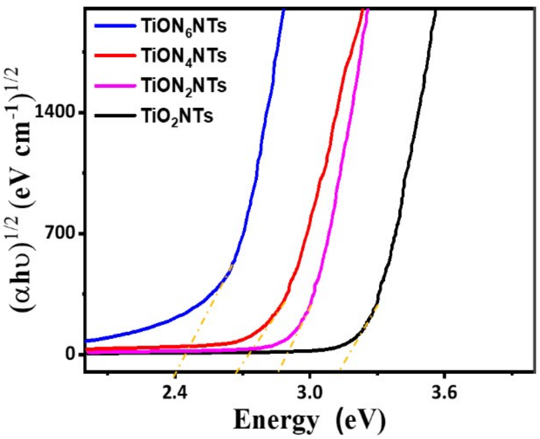

Figure S2 Tauc plot of ammonia annealed $TiON_6NTs$, $TiON_4NTs$, and $TiON_2NTs$ relative to air annealed TiO_2NTs.

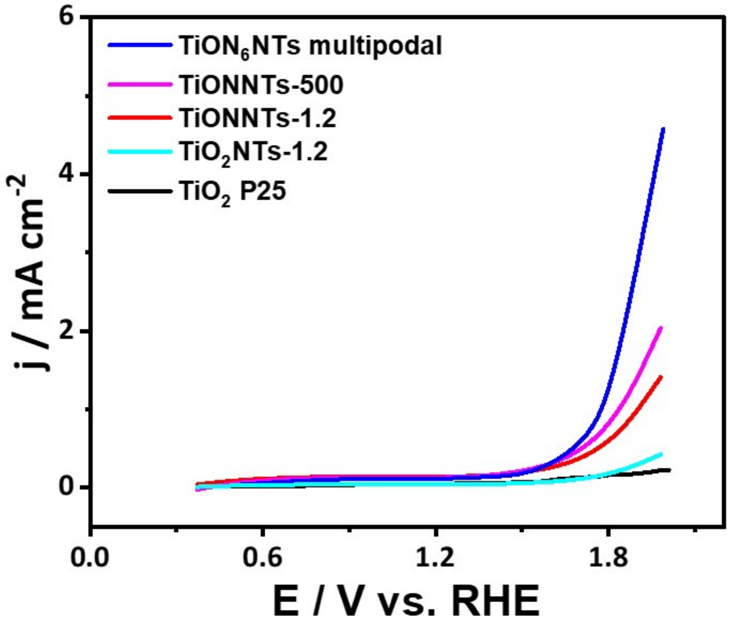

Figure S2 LSV of water oxidation on different photocatalysts with different lengths and shapes in an aqueous solution of 0.1 M KOH at a scan rate of 10 mV s^{-1} at room temperature under light illumination.

$TiON_6NTs$ is multipodal (80 nm length), TiONNTs-1.2 is ammonia annealed TiO_2NTs (1.2 µm length), TiONNTs-500 is ammonia annealed TiO_2NTs (500 nm length), TiO_2NTs-1.2 is nitrogen-free (1.2 µm length), and TiO_2 is commercial TiO_2 nanoparticles (spherical 5-8 nm). $TiON_6NTs$, TiONNTs-1.2, TiONNTs-500, and TiO_2NTs-1.2, were used as working electrode, while TiO_2 nanoparticles were deposited on indium tin oxide substrate with the same area as other NTs foils.

TiONNTs-1.2 and TiO_2NTs-1.2 were prepared using our previous reported method[1]. Meanwhile, the same method was used to fabricate TiONNTs-500 but with decreasing the anodic oxidation time to 80 min. TiONNTs-1.2 and TiONNTs-500 were annealed under ammonia for 6 h.

Table S2 The donner densities band potential obtained from Mott-Schottky plots

		TiO_2NTs	$TiON_2$NTs	$TiON_4$NTs	$TiON_6$NTs
Light	R_1 (Ω.cm^2)	192.3	167.1	128.1	112.4
	CPE_1 Y_{o1} x 10^{-6} (s^n ohm^{-1} cm^{-2})	1036	1345	2093	2467
	n_1	0.874	0.743	0.719	0.667
	R_2 (Ω.cm^2)	2115	2012	1423	1264
	CPE_2 Y_{o2} x 10^{-6} (s^n ohm^{-1} cm^{-2})	93.51	99.13	157.2	213.7
	n_2	0.613	0.597	0.572	0.556

Table S3 The donner densities band potential obtained from Mott-Schottky plots

	N_d / cm^{-3}	E_{FB} / V vs. RHE
TiO_2NTs	1.68E+19	0.89
$TiON_2$NTs	8.24E+18	1.16
$TiON_4$NTs	4.15E+18	-1.57
$TiON_6$NTs	3.54E+18	-1.94

References

1. K. Eid, K. A. Soliman, D. Abdulmalik, D. Mitoraj, M. H. Sleim, M. O. Liedke, H. A. El-Sayed, A. S. AlJaber, I. Y. Al-Qaradawi and O. M. Reyes, *Catal. Sci. Technol.*, 2020, **10**, 801-809.