Supporting Information for

Controlled Thermal Nitridation Resulting in Improved Structural and Photoelectrochemical Properties from Ta_3N_5 Nanotubular Photoanodes

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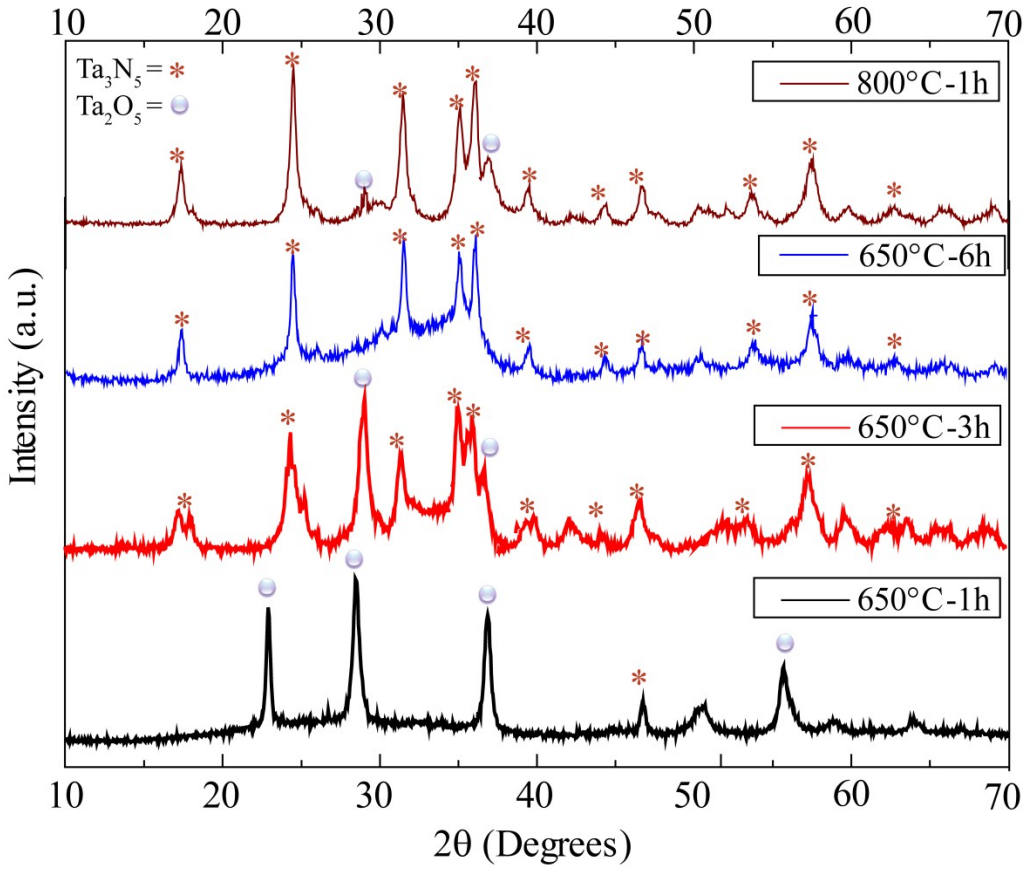

Fig. S1 XRD patterns of the samples prepared at 650°C and 800°C of nitridation.

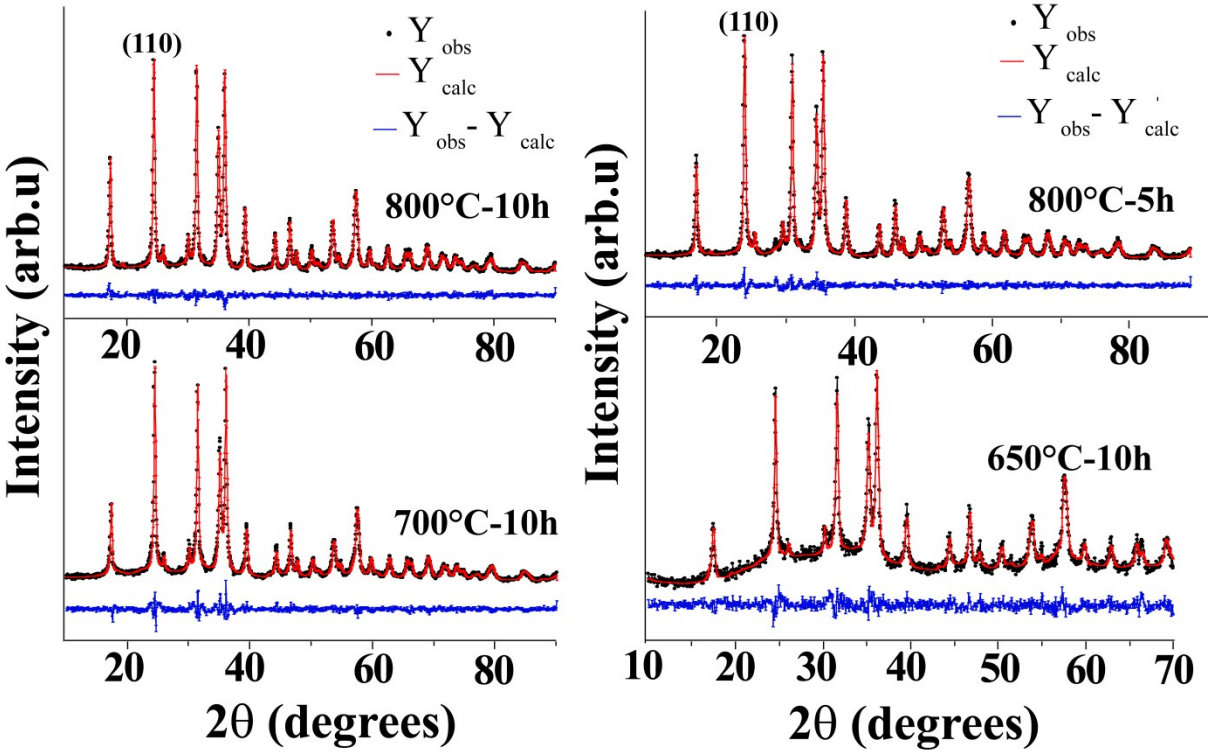

Fig. S2 Rietveld refinement profile for Ta_3N_5 NTs; (•) observed Y_{obs}, (red solid lines) calculated Y_{calc}, (blue solid lines) Y_{obs} - Y_{calc} difference of phase.

Table S1 Estimated geometrical dimensions of Ta_3N_5 nanotubes as a function of nitridation temperature and time.*

Sample	Wall thickness/nm*	Diameter/nm*	Length/μm†
As-Anodized	34	130	1.60
650°C-10h	34	128	1.58
700°C-10h	33	125	1.52
750°C-10h	32	110	1.40
800°C-2h	33	120	1.50
800°C-3h	30	100	1.40
800°C-5h	28	95	1.35
800°C-10h	28	90	1.20
850°C-3h	28	90	1.25
900°C-3h	25	75	1.10
900°C-10h	-	-	-
1000°C-3h	-	-	-

* *Wall thicknesses and Diameters of the NTs were calculated from TEM images taken from different locations.*

† *Lengths of the NTs were calculated from SEM images.*

Table S2 Percentages of vacancies (vac), occupancies (occ), substitutions (sub) in the Wyckoff positions, cell parameters and agreement factors deduced from the Rietveld refinements.

Sample	Ta1 (4c)	Ta2 (8f)	N1 (4c)	N2/O2 (8f)	N3 (8f)	R_{bragg}-factor	R_f-factor	Chi^2	*a* *b* *c*
650°C-10h	97.98 occ 2.02 vac	99.89 occ 0.11 vac	71.33 occ 28.67 vac	0 – occ 100 subs	99.98 occ 0.02 vac	6.19	4.06	1.43	3.88516 10.16713 10.24118
700°C-10h	94.60 occ 5.40 vac	99.97occ 0.03 vac	69.71 occ 30.29 vac	37.10 occ 62.90 sub	100 occ 0 – vac	3.33	1.94	1.99	3.89520 10.19483 10.26891
800°C-5h	93.87 occ 6.128 vac	98.32 occ 1.68 vac	94.40 occ 5.60 vac	66.01 occ 33.99 sub	100 occ 0 – vac	2.70	1.45	1.51	3.89962 10.20950 10.27345
800°C-10h	94.58 occ 5.42 vac	98.39 occ 1.61 vac	95.03 occ 4.97 vac	80.01 occ 19.99 sub	100 occ 0 – vac	4.14	2.41	1.37	3.89325 10.20868 10.26509

Table S3 (Photo)electrochemical parameters of Ta_3N_5 NTs obtained under various conditions of temperatures and nitridation times.*

Sample	V_{oc}(dark)V vs Ag/AgCl	V_{oc} (AM 1.5) V vs Ag/AgCl	I_{ph} (0.64V vs RHE) 0 V vs Ag/AgCl	I_{ph} (1.23V vs RHE) 0.6 V vs Ag/AgCl
650°C-10h	0.10	–0.32	70 µA	180 µA
700°C-10h	0.10	–0.32	151 µA	328 µA
750°C-10h	0.11	–0.36	251 µA	710 µA
800°C-1h	0.08	–0.26	107 µA	98 µA
800°C-02h	0.10	–0.34	251 µA	556 µA
800°C-05h	0.10	–0.36	940 µA	2.6 mA
800°C-10h	0.11	–0.33	280 µA	2.4 mA
900°C-10h	0.11	–0.35	1.8 mA	3.9 mA
1000°C-3h	0.10	–0.34	600 µA	2.8 mA

* To calculate I_{ph} the current in dark was subtracted.