

Supporting information

## Understanding the Morphological Evolution of Anodic Tantalum Oxide Nanostructures in Acidic Medium

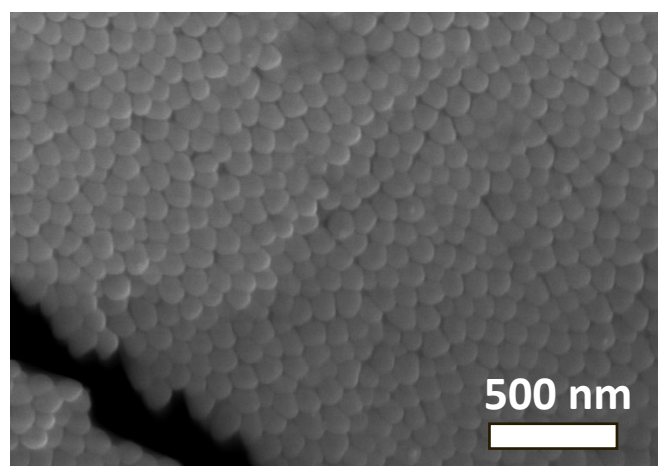
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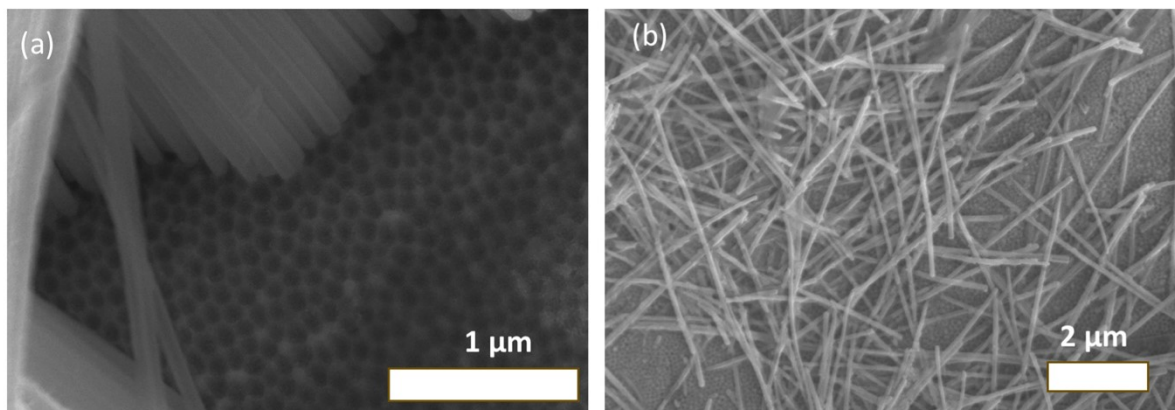
<sup>2</sup> Faculty of Physics and Applied Computer Science, AGH University of Krakow, Mickiewicza 30, 30-059 Krakow, Poland

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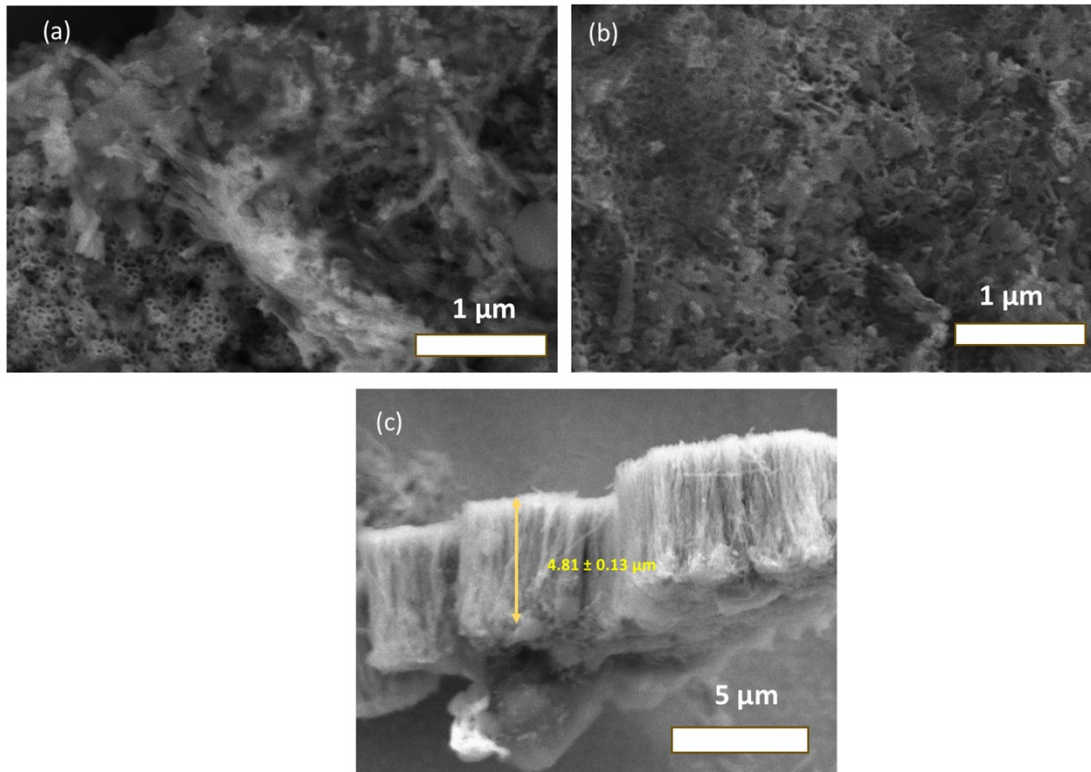
Email: biswaranjan.mohapatra@uj.edu.pl; sulka@chemia.uj.edu.pl



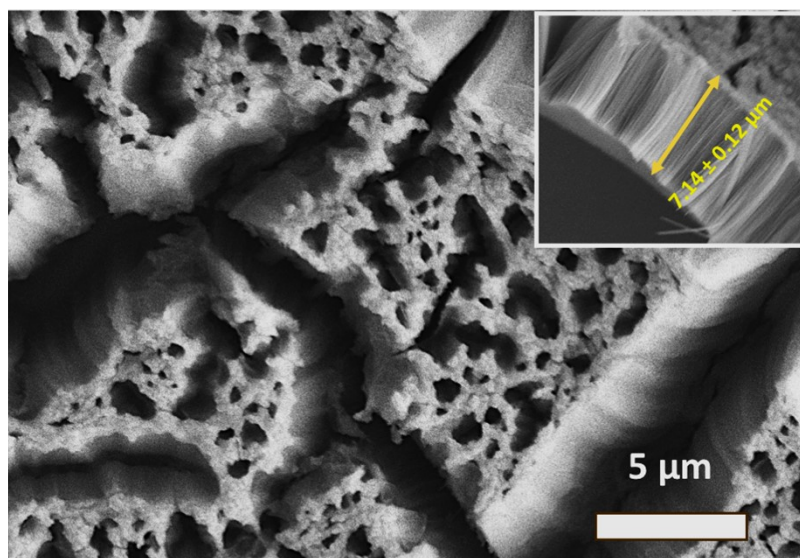
**Figure S1:** SEM image showing the bottom layer of the ATO nanotubes produced at 40 V for 15 min in electrolyte containing 90 ml H<sub>2</sub>SO<sub>4</sub> (95 wt.%) + 6 ml HF (40 wt.%) + 4 ml H<sub>2</sub>O at 23°C.



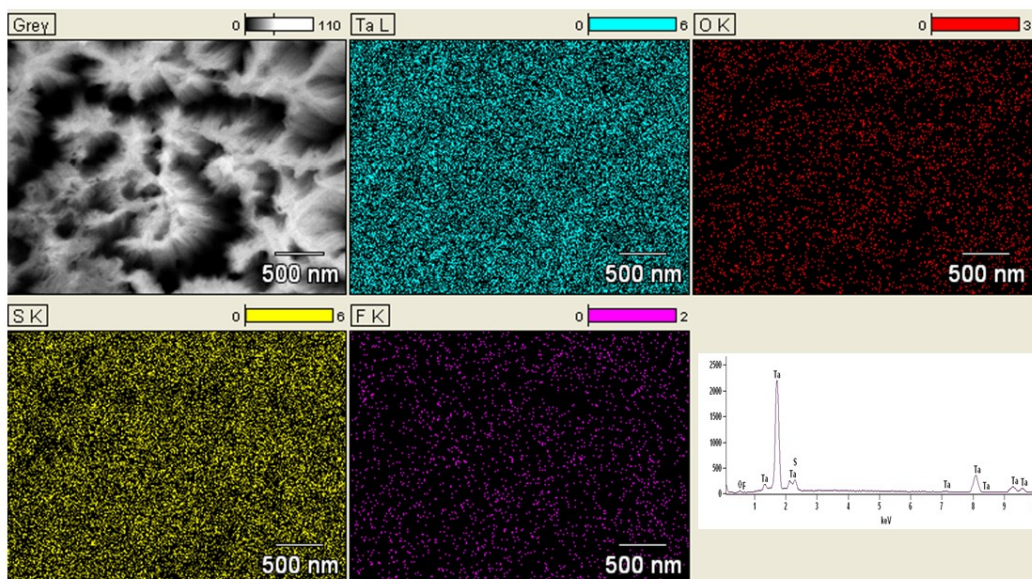
**Figure S2:** SEM image showing (a) the nano-dimple layer on the Ta surface after the removal of ATO nanotubes (b) the freestanding ATO nanotubes produced at 40 V for 4 min in electrolyte containing 90 ml H<sub>2</sub>SO<sub>4</sub> (95 wt.%) + 6 ml HF (40 wt.%) + 4 ml H<sub>2</sub>O at 23°C.



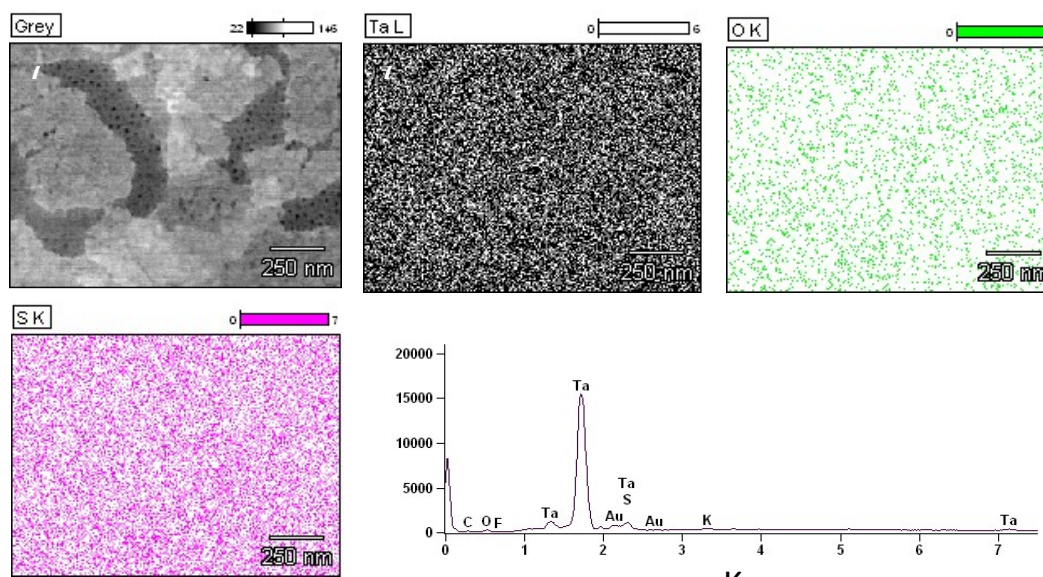
**Figure S3.** Top SEM image view of ATO structures produced at 80 V for (a) 2 min and (b) 4 min in electrolyte containing 90 ml H<sub>2</sub>SO<sub>4</sub> (95 wt.%) + 6 ml HF (40 wt.%) + 4 ml H<sub>2</sub>O at 23°C. (C) SEM side view image for the ATO structures produced at 80 V for 2 min.



**Figure S4.** Top SEM image view of ATO structures produced at 15 V for 6 hr in electrolyte containing 90 ml H<sub>2</sub>SO<sub>4</sub> (95 wt.%) + 6 ml HF (40 wt.%) + 4 ml H<sub>2</sub>O at 23°C.



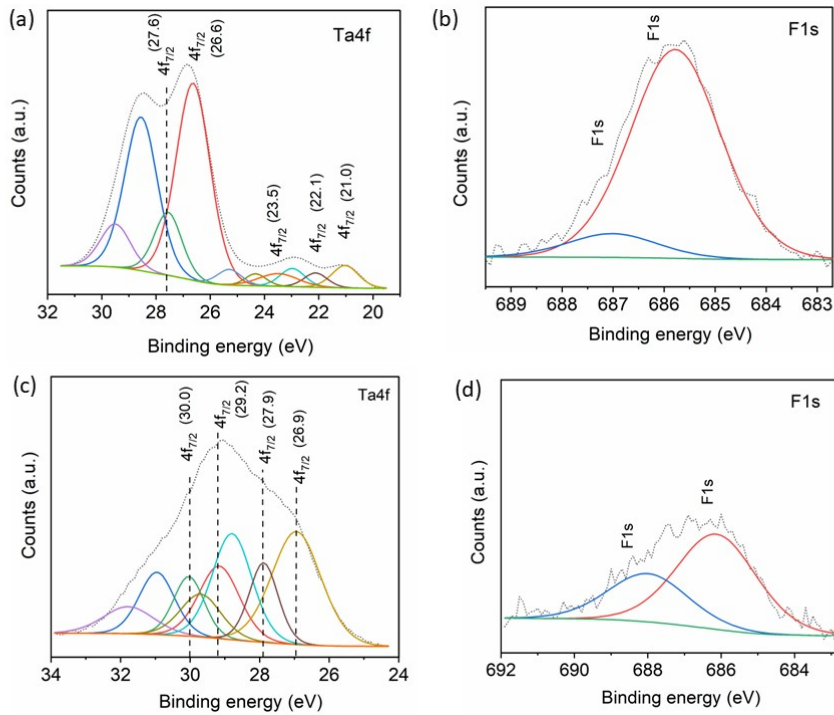
**Figure S5:** Surface morphology, EDS elemental mapping, and corresponding EDX spectrum for self-organized ATO nanotubes formed at 40 V for 4 min in the electrolyte containing 90 ml H<sub>2</sub>SO<sub>4</sub> (95 wt.%) + 6 ml HF (40 wt.%) + 4 ml H<sub>2</sub>O at 23°C.



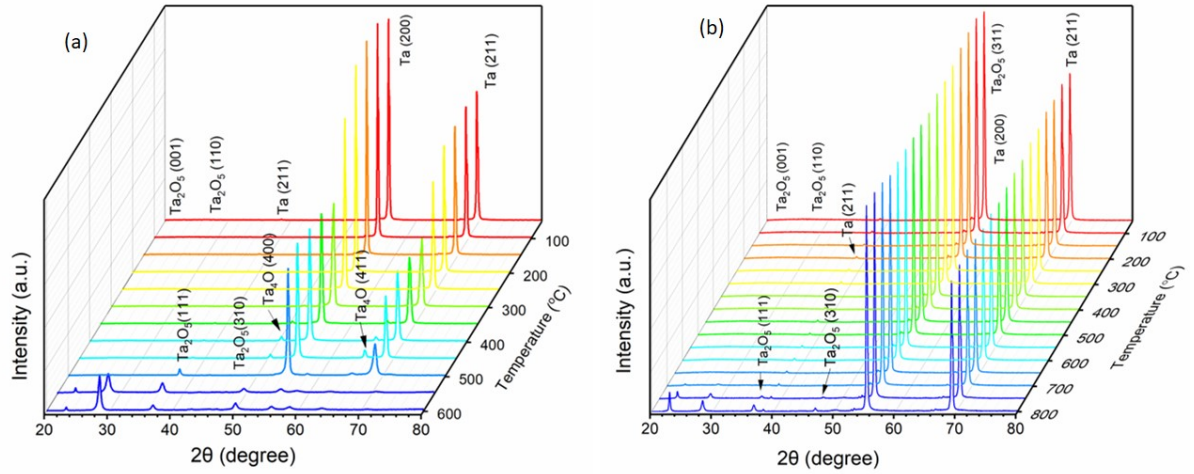
**Figure S6:** Surface morphology, EDS elemental mapping, and corresponding EDX spectrum for self-organized ATO nanotubes formed at 15 V for 15 min in the electrolyte containing 90 ml H<sub>2</sub>SO<sub>4</sub> (95 wt.%) + 6 ml HF (40 wt.%) + 4 ml H<sub>2</sub>O at 23°C.

Samples	Chemical composition (at. %)			
	Ta4f	O1s	S2p	F1s
ATO-15	17.9	64.8	15.9	1.5
ATO-30	18.4	64.4	15.9	1.3
ATO-40	14.5	66.2	18.3	1.0

**Table S1.** Chemical composition of ATO produced at 15 (15 min), 30 (4 min) and 40 V (4 min) derived from the XPS results. The samples are abbreviated as ATO-15, ATO-30 and ATO-40, respectively.



**Figure S7.** Deconvoluted (a) Ta4f and (b) F1s XPS spectra of sample ATO-15. Deconvoluted (c) Ta4f and (d) F1s XPS spectra of sample ATO-40.



**Figure S8:** Temperature programmed XRD spectra in (a) air and (b) argon atmosphere for ATO structures formed at 40 V for 4 min in a 100 ml of electrolyte containing 90 ml  $\text{H}_2\text{SO}_4$  (95 wt.%) and a 6:4 v/v of HF:H $_2\text{O}$  ratio.