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Electronic Supplementary Information

Ultrathin Anatase Nanosheets with High Energy Facets Exposed and Related Photocatalytic Performances

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1. Experimental details

Synthesis of Exfoliated Titanate Nanosheets

The exfoliated titanate nanosheets were prepared through a former reported method.[1] In detail, the starting material of $Cs_{0.7}Ti_{1.825}O_4$, was prepared by repeated calcination of the stoichiometric molar ratio mixture of TiO_2 and Cs_2CO_3 twice at 1073 K for 20 h. The obtained $Cs_{0.7}Ti_{1.825}O_4$ powders were treated with 1.0 M HCl for 4 days to achieve the corresponding protonated derivative $H_{0.7}Ti_{1.825}O_4{\cdot}xH_2O$. The resulted $H_{0.7}Ti_{1.825}O_4{\cdot}xH_2O$ was exfoliated by tetrabutylammonium hydroxide (TBAOH , equal molar to proton) at room temperature under weak stirring condition for more than 10 days. After exfoliation, the obtained titanate colloid was stood for more than 15 days to sedimentate and remove the unexfoliated titanate. The final pale blue colloid was identified as exfoliated single layer titanate nanosheets (TiONS). The morphology and thickness of TiONS were confirmed by TEM and AFM in Fig. S1.

Synthesis of uniform SiO_2 nanoparticles

Typically, 0.35 g lysine was dissolved in 325.0 g distilled water. Then 25.0 g Tetraethylorthosilicate (TEOS) was added into the solution of lysine under continuous stirring at 333 K for 48 h to undergo a catalytic hydrolysis. After complete hydrolysis, the obtained sol was collected for the following use. The morphology and the size of the obtained silica nanoparticles were confirmed by transmission electron microscopy (Fig. S2).

Synthesis of TS-Xs

Hybrid TS-Xs were prepared through a freeze-drying method. The TiONS and SiO_2 sol were mixed according to various T/S mass ration as shown in Table S1 and stirred for more than 30 min. The mixed sol was put into freezing dryer (233 K ~ 223 K) to solidification for 24 h, then the frozen sample was dried in freezing dryer. The resulted cotton like solids were calcined at 773 K in air for 5 h. The color of the solids changed from white to black and final to pure white.

Table S1. The list of the prepared samples.

Sample	**TS-1**	**TS-2**	**TS-3**	**TS-4**	**TS-5**	**TS-6**
m(TiO_2) / m(SiO_2)	10/1	5/1	2.5/1	1/1	1/2.5	1/5

Characterizations

Powder X-ray diffraction (XRD) patterns were recorded on a Phillips X'Pro diffractometer using Cu Kα radiation (λ = 1.5418 Å) at 40 kV and 40 mA. Transmission electron microscopy (TEM) images were taken with a JEOL JEM-1011 Electron Microscope at an accelerating voltage of 100 kV. High resolution transmission electron microscopy (HRTEM) measurements were conducted using a JEOL JEM-2100 instrument at an accelerating voltage of 200 kV. Scanning electron microscopy (SEM) measurements were conducted with a Hitachi S-4800 instrument. AFM The nitrogen sorption isotherms were measured at liquid-nitrogen temperature on an ASAP 2020 apparatus. The specific surface area was calculated with the Brunauer–Emmett–Teller (BET) equation. The pore size distribution was derived from the desorption branches of the isotherms by Barrett–Joyner–Halenda (BJH) method.

Photocatalytic performance test

A desired amount of catalyst (containing 5 mg TiO_2) was suspended in 400 mL RhB aqueous solution (2×10^{-5} M) in a quartzose beaker. A high pressure mercury lamp (125 W) was used as the light source. Before the light was turned on, the suspension was magnetically stirred well in the dark at room temperature for more than 40 min until an adsorption–desorption equilibrium was finally established. When the light was turned on, at given time intervals, approximately 8.0 ml of the reaction suspension was sampled and separated by high-speed centrifugation. The absorption spectrum of the filtrate was measured on a UV-visible spectrometer, and the concentration of RhB was determined by monitoring the change in the absorbance maximized at about 554 nm.

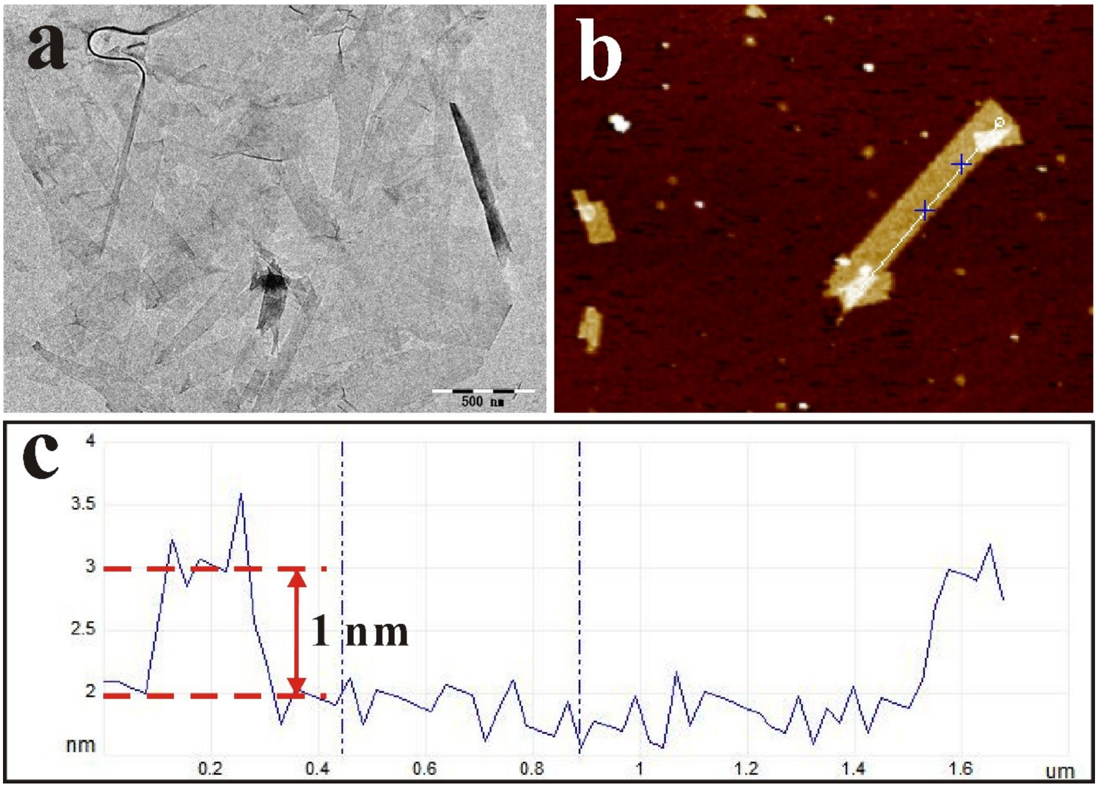

Fig. S1. (a) TEM image of the exfoliated TiONS, (b) AFM image of TiONS, (c) AFM profiles along line in (b).

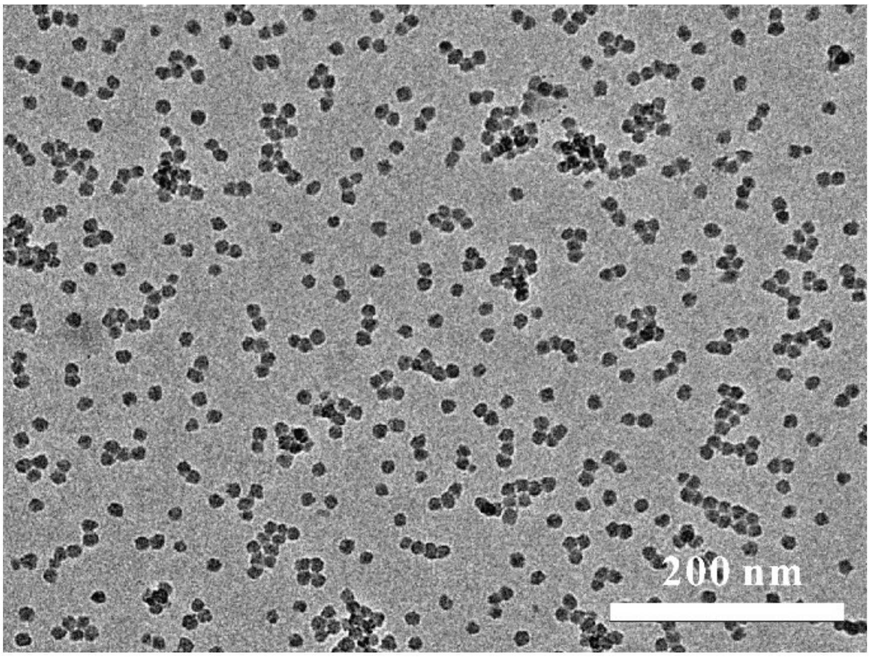

Fig. S2. TEM image of the as-made silica nanoparticles.

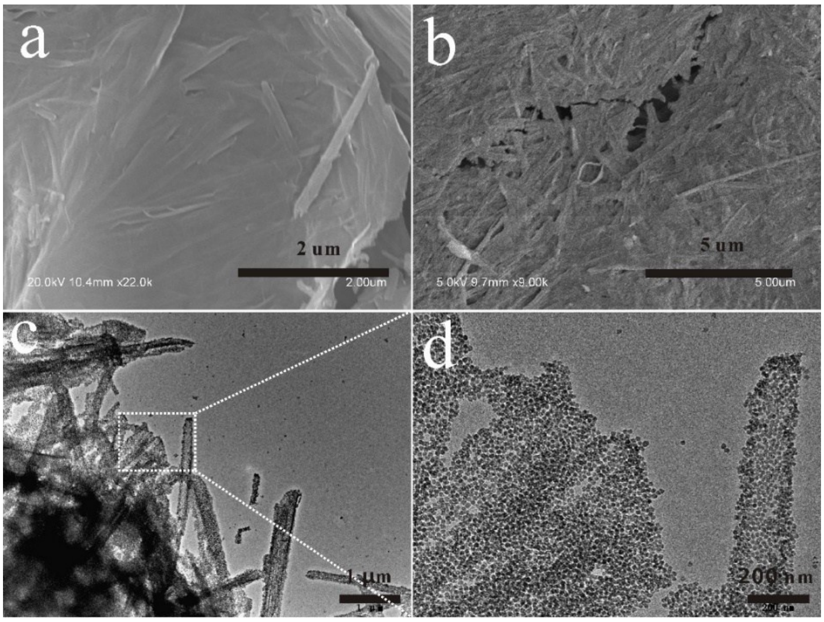

Fig. S3 (a) SEM image of the exfoliated TiONS; (b) SEM images of TS-5; (c) TEM image of TS-5; (d) magnified TEM image of white rectangle region in (c).

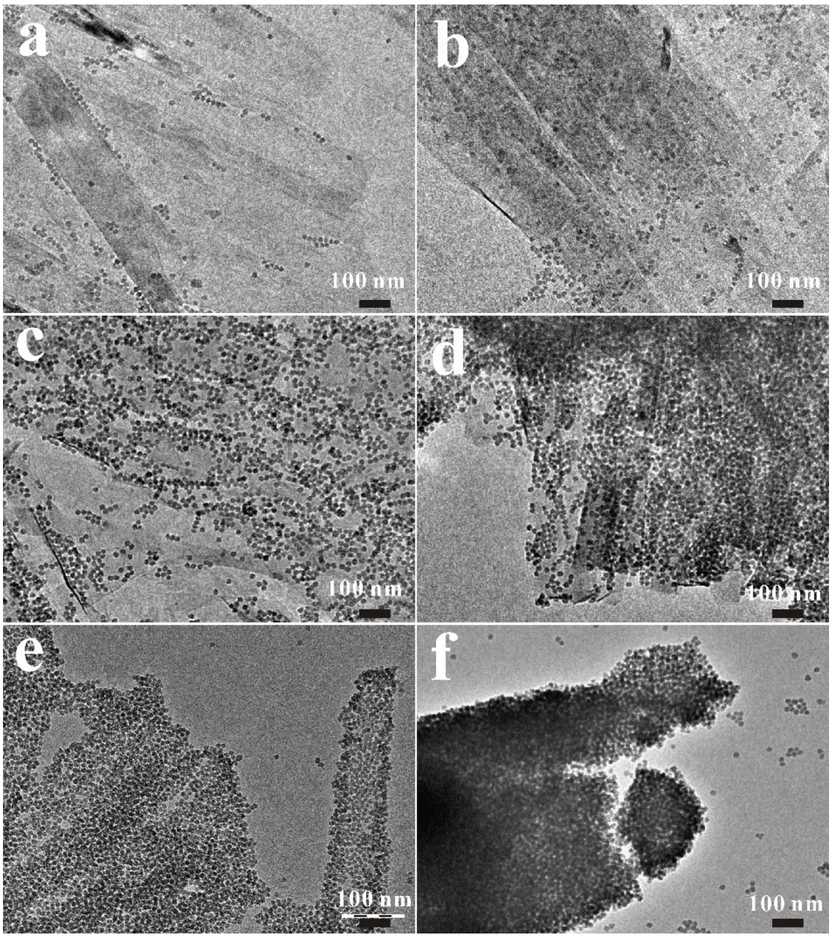

Fig. S4. TEM images of (a) TS-1, (b) TS-2, (c) TS-3, (d) TS-4, (e) TS-5 and (f) TS-6.

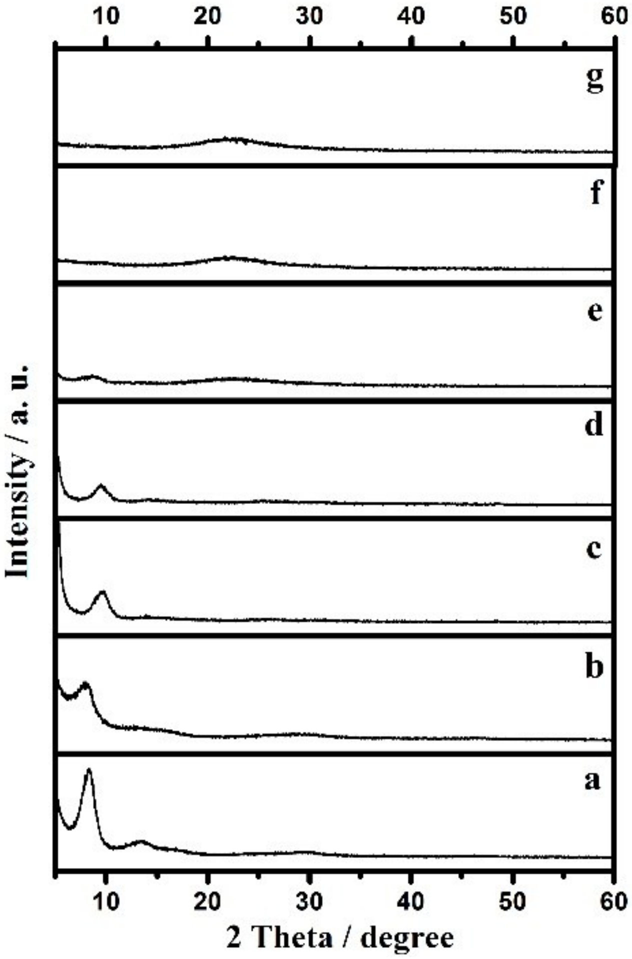

Fig. S5. XRD patterns of (a) dried TiONS, (b) TS-1, (c) TS-2, (d) TS-3, (e) TS-4, (f) TS-5 and (g) TS-6.

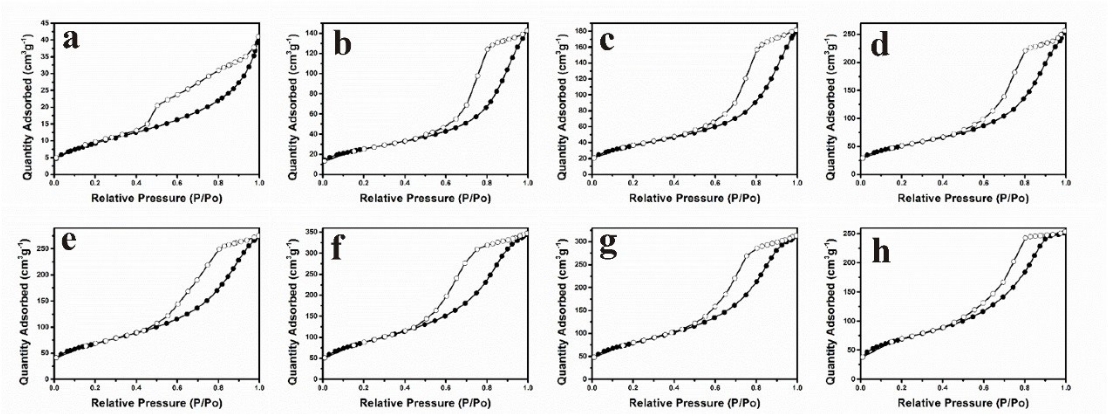

Fig. S6. Nitrogen sorption isotherm of each sample: (a) Calcined pure TiONS, (b)TS-1-C, (c) TS-2-C, (d) TS-3-C, (e) TS-4-C, (f) TS-5-C, (g) TS-6-C and (h) Calcined SiO_2 NPs.

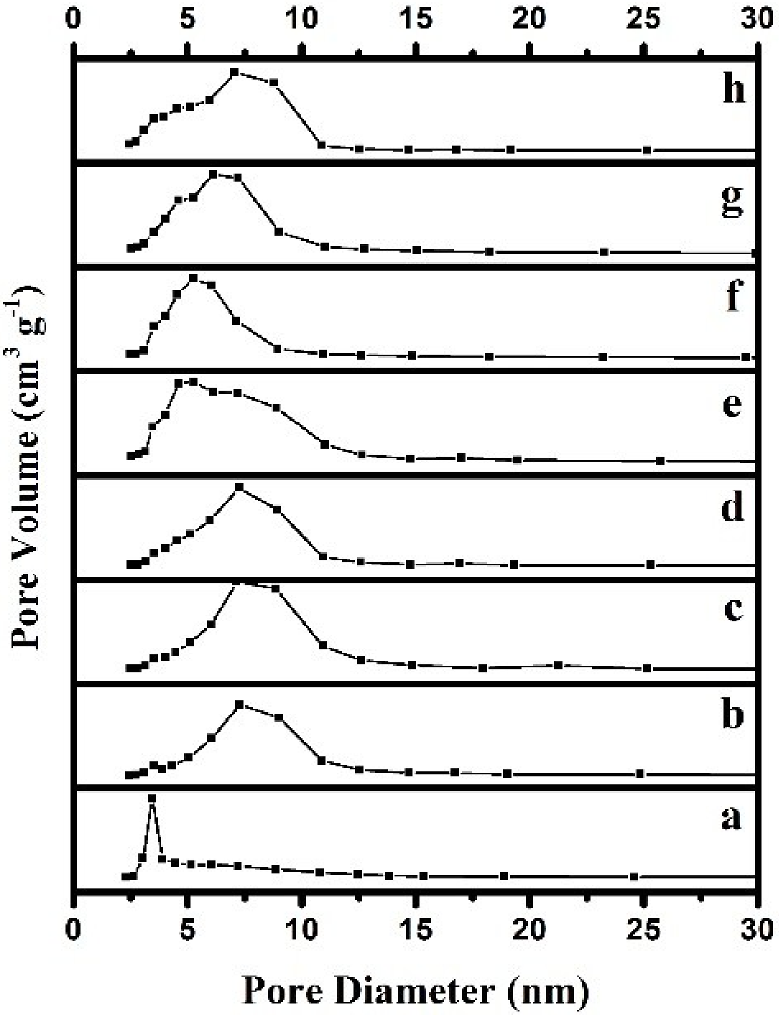

Fig. S7. Pore distribution of each sample: (a) Calcined pure TiONS, (b) TS-1-C, (c) TS-2-C, (d) TS-3-C, (e) TS-4-C, (f) TS-5-C, (g) TS-6-C and (h) Calcined SiO_2 NPs.

Table S2. The textual parameters of the Calcined TiONS, TS-Xs and Calcined silica nanoparticles.

Sample	S_{BET} ($m^2 g^{-1}$)	V_{total} $cm^3 g^{-1}$)	Pore size (nm)	S_{Micro} ($m^2 g^{-1}$)	$S^a{}_{Exter}$ ($m^2 g^{-1}$)
Calcined TiONS	35	0.05	6.24	0	35
TS-1-C	93	0.21	9.00	0	93
TS-2-C	132	0.27	8.03	3	129
TS-3-C	185	0.37	7.97	2	183
TS-4-C	247	0.41	6.66	16	231
TS-5-C	318	0.52	6.56	5	313
TS-6-C	284	0.47	6.63	24	260
Calcined SiO_2 NPs	251	0.39	6.19	14	237

a: S_{Exter} represents the surface area outside the microporosity.

References:

1. T. Sasaki and M. Watanabe, *J. Am. Chem. Soc.*, 1998, **120**, 4682-4689.