## One-step synthesis of ultrathin nanobelts-assembled urchin-like anatase TiO<sub>2</sub> nanostructures for highly efficient photocatalysis

Xin Yu<sup>a1</sup>, Zhenhuan Zhao<sup>b1</sup>, Jian Zhang<sup>c</sup>, Weibo Guo<sup>d</sup>, Linlin Li<sup>a\*</sup>, Hong Liu<sup>ac\*</sup>, Zhong Lin Wang<sup>a\*</sup>

<sup>a</sup> Beijing Institute of Nanoenergy and Nanosystems, Chinese Academy of Sciences;

National Center for Nanoscience and Technology (NCNST), Beijing, 100083, P. R.

China

<sup>b</sup> Institute of Fundamental and Frontier Sciences, University of Electronics Science and Technology of China, Chengdu, 610054, P. R. China

<sup>c</sup> Universite de lyon, ECL, INSA-Lyon, UCBL, CPE, CNRS, INL, UMR 5270, 36 avenue Guy de Collongue, 69134 Ecully cedex France

<sup>d</sup> Shandong provincial key laboratory of detection technology for tumor markers, college of chemistry and chemical engineering, Linyi university, Linyi 276005, P. R. China

<sup>e</sup> State Key Laboratory of Crystal Materials Shandong University, Jinan, 250100, P. R. China

\*Corresponding author. E-mail addresses: lilinlin@binn.cas.cn, hongliu@sdu.edu.cn, zlwang@gatech.edu



Fig. S1 TEM images of the urchin-like  $TiO_2$  shell structure synthesized with 1.0 g urea.



Fig. S2 XPS fully scanned spectrum of the urchin-like anatese TiO<sub>2</sub>.



Fig. S3 SEM images of the  $TiO_2$  nanorod structure with 0.1 g urea.



Fig. S4 TEM images of the  $TiO_2$  sphere with solid core.

Form the TEM image in Fig. S4, it could be found that sample had solid structure with surface assembled nanobelts.



Fig. S5 SEM images of the TiO<sub>2</sub> samples with different amount of diethylene glycol.
(a) 40 ml deionized water without diethylene glycol, (b) 30 ml deionized water, 10 ml diethylene glycol, (c) 20 ml deionized water, 20 ml diethylene glycol.

From the SEM images of the  $TiO_2$  samples in Fig. S5, it can be see that without the diethylene glycol or with less diethylene glycol compared to the 10 ml deionized water and 30 ml diethylene glycol it cannot form the  $TiO_2$  ultrathin nanobelts and urchin-like structures. The  $TiO_2$  nanoparticles agglomerated into big blocks.



Fig. S6 SEM images of the TiO<sub>2</sub> sample with NH<sub>4</sub>OH (1 ml wt 25%) instead of urea.

From the Fig. S6 it can be see that when used  $NH_4OH$  instead of urea the  $TiO_2$  formed the ultrathin nanobelts, but the nanobelts agglomerated into blocks. So we can provenly concluded that it was  $CO_2$  acted as the soft templates for providing

aggregation centers of TiO<sub>2</sub> seeds to form hollow structure.

**Table S1** The TiO<sub>2</sub> yield of the different synthesis condition after 4 h reaction time. 0.35 g  $K_2TiO(C_2O_4)_2$  was used as the titanium source. It can be seen that the production of TiO<sub>2</sub> increase with the urea increasing.

| Urea                   | 0 g   | 0.1 g | 0.5 g | 1.0 g |
|------------------------|-------|-------|-------|-------|
| TiO <sub>2</sub> Yield | 0.015 | 0.043 | 0.061 | 0.075 |
| [g]                    |       |       |       |       |



**Fig. S7** Photocatalytic degradation of MO without any catalyst and in the presence of photocatalysts but in dark.

In the Fig. S7, to check if MO can be degraded under light irradiation without photocatalyst, 20 mg/L of MO solution was irradiated under UV and visible light,

respectively, and the degradation rate of MO were checked. The result showed that there was no decrease of MO concentration without photocatalyst under UV light irradiation. And these results indicated that the adsorption-desorption equilibrium of MO had been established within 5 min in the dark. And there was no appreciable degradation of MO after 30 min in the absence of photocatalysts.



Fig. S8 Comparison of the photocatalytic activities of the  $TiO_2$  samples synthesized with the different concentration of urea.



**Fig. S9** photographs of Degussa P25 and urchin-like TiO<sub>2</sub> following sedimentation for 60 min.



Fig. S10 SEM images of the (a)  $TiO_2$  nanobelt (b) A- $TiO_2$  (c) P25 (d) urchin-like anatase  $TiO_2$ .



Fig. S11 XRD patterns of the  $TiO_2$  nanobelt, commercial A- $TiO_2$ , P25 and urchin-like anatase  $TiO_2$ .

The crystallite size of the  $TiO_2$  nanoparticles, *D*, was calculated by using the Debye-Scherre formula from the major diffraction peak of the corresponding  $TiO_2$ . The Debye Scherrer formula is as follows:

$$D = \frac{K \lambda}{\beta \cos^{\mu}(\theta)}$$

Where K is Scherrer constant (K = 0.89),  $\lambda$  is the X-ray wavelength used in XRD (1.5418 Å),  $\beta$  is the pure diffraction broadening of a peak at half-height and  $\theta$  is the Bragg angle. The average diameter of the TiO<sub>2</sub> nanoparticles calculated is 17 nm. And the other samples had a relatively larger half peak width with the diameter 92 nm, 35 nm, 39 nm for the TiO<sub>2</sub> nanobelt, A-TiO<sub>2</sub> and P25, respectively.



Fig. S12 Nitrogen adsorption-desorption isotherms of the different samples. (a) 0 g urea (b) 0.1 g urea (c) 0.5g urea (d) 1.0 g urea (e) commercial A-TiO<sub>2</sub> (f) Degussa P25.

 Table S2 Brunauer–Emmett–Teller specific surface area of the different samples. (a)

 0 g urea (b) 0.1 g urea (c) 0.5 g urea (d) 1.0 g urea (e) commercial anatase TiO<sub>2</sub> (f)

 Degussa P25.

| Urea                     | 0 g     | 0.1 g   | 0.5 g    | 1.0 g    | P25     | A-TiO <sub>2</sub> |
|--------------------------|---------|---------|----------|----------|---------|--------------------|
| BET [cm <sup>2</sup> /g] | 55.6607 | 50.8008 | 162.1970 | 170.9779 | 53.3879 | 55.5366            |