

## Supporting Information

# **Hierarchical Nanostructures of $\gamma$ -TaON Flower for Enhanced Visible Light Photocatalytic Activities**

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## **EXPERIMENTAL SECTION**

### **Synthesis**

Ethanol, hydrogen peroxide and hydrofluoric acid (Caution! Hydrofluoric acid is highly corrosive and it must be handled carefully.) were supplied by Sinopharm Chemical Reagent Beijing Co., Ltd (SCRB). Tantalum powders (99.9%) were supplied from Aladdin. Ammonia gas was supplied by Haipu Gas Co., Ltd. All materials were used as supplied. Deionized (DI) water was used in all experiments. At first, tantalum oxide three-dimensional nanostructures are prepared by a hydrothermal process. In a typical synthesis procedure, stoichiometric tantalum powder was added to a certain amount of hydrofluoric acid (0.1 M) and hydrogen peroxide (2.0 M) aqueous solution, which was loaded into a Teflon-lined cylindrical autoclave. Then the sealed autoclave was heated at 240°C for several hours in a box furnace. The final precipitates were separated, washed with ethanol and DI water for three times and then dried in oven at 60 °C for 12 h to obtain the final products of composite powders. After that, the  $\gamma$ -TaON samples were prepared by nitridation at 850 °C for 5 h under the flow of ammonia gas (20 mL min<sup>-1</sup>) through a conical flask containing 100 mL deionized water at 30 °C.

### **Characterization**

The obtained products were characterized with powder X-ray diffraction (XRD, MAC Science Co. Ltd Japan) using Cu K $\alpha$  ( $\lambda = 0.1546$  nm) and XRD patterns were obtained for 10-90° 2 $\theta$  by step scanning with a step size of 0.02°. The morphology and size of the resultant powders were characterized by a Zeiss Ultra 55 field-emission scanning electron microscope (SEM) associated with X-ray energy-dispersive spectrometer (EDX). Transmission electron microscopy (TEM) images

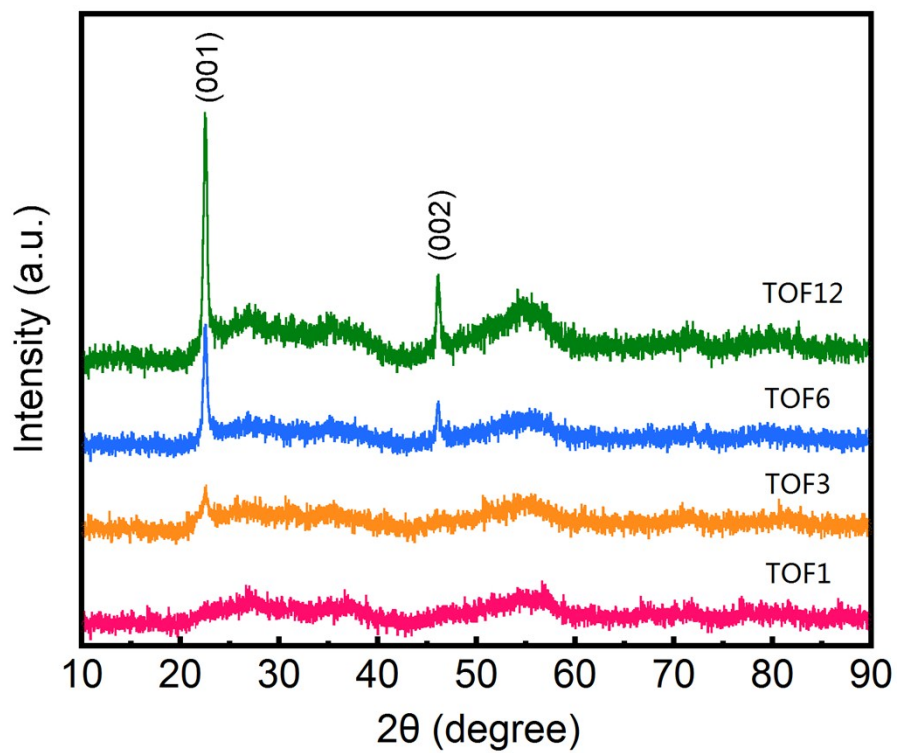
were captured on the transmission electron microscopy (TEM, JEM-2010) at an acceleration voltage of 200 kV. The optical properties of the samples were analyzed by UV-vis diffuse reflectance spectroscopy (UV-vis DRS) using a UV-vis spectrophotometer (UV-2550, Shimadzu). Fine BaSO<sub>4</sub> powder is used as a standard for baseline and the spectra are recorded in a range 190-900 nm. The chemical states of the sample were determined by X-ray photoelectron spectroscopy (XPS) in a VG Multilab 2009 system (UK) with a monochromatic Al K $\alpha$  source and charge neutralizer.

### **Photocatalytic Tests**

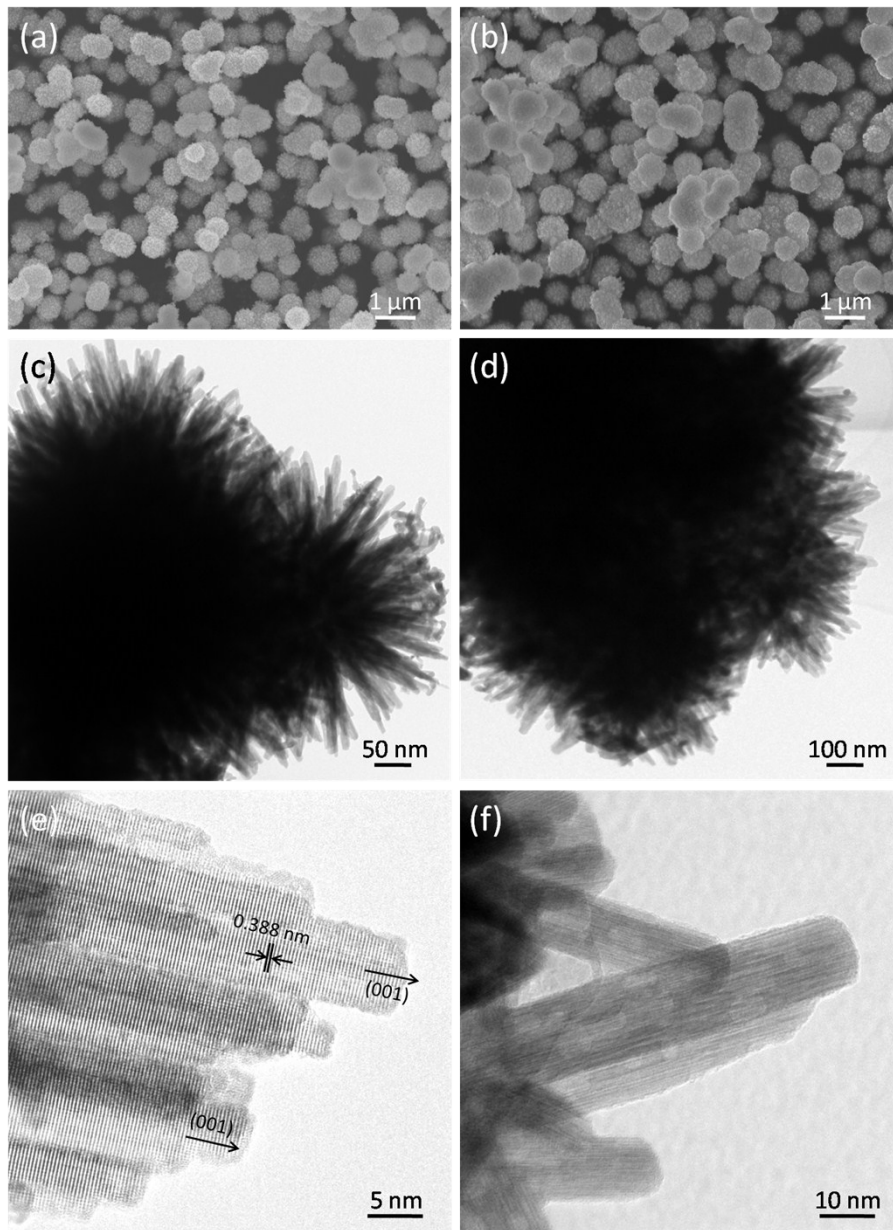
Photocatalytic hydrogen production was carried out in an air free closed gas circulation system reaction cell made of quartz. The total cylindrical volume of the cell was 250 mL. An optically polished piece of quartz glass was fused on top of the cell to minimize light scattering. Hydrogen evolution was detected using a gas chromatograph (Beijing, GC-3240, TCD, Ar carrier), which was connected to a gas-circulation line. The reaction was initiated by irradiation with a 300 W xenon lamp fitted with a cut off filter ( $\lambda > 420$  nm). The whole system, including the photocatalyst, was flushed with Ar at 100 mL min<sup>-1</sup> for 1 h to remove any trace of air (including nitrogen and oxygen) before any photocatalytic reaction was carried out. During the process, agitation of the solution ensured uniform irradiation of the suspensions. Quantum efficiency (QE) was measured under identical photocatalytic reaction conditions with irradiation light at  $\lambda = 420$  nm by using a 300 W xenon lamp and combined band pass and cut-off filters. The number of incident photons was also calibrated with a Si photodiode. The QE was calculated according to the following equation:

$$\begin{aligned} \text{QE (\%)} &= \frac{\text{The rate of reacted electrons}}{\text{The rate of incident photons}} \times 100 \\ &= \frac{\text{The rate of evolved H}_2 \text{ molecules} \times 2}{\text{The rate of incident photons}} \times 100 \end{aligned} \quad (1)$$

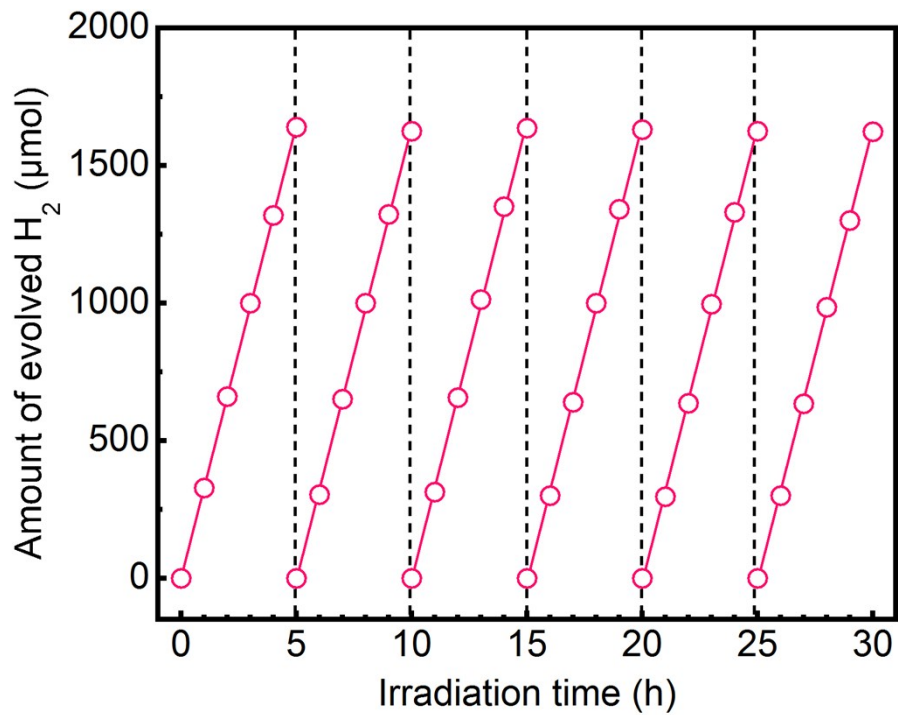
## Additional Results



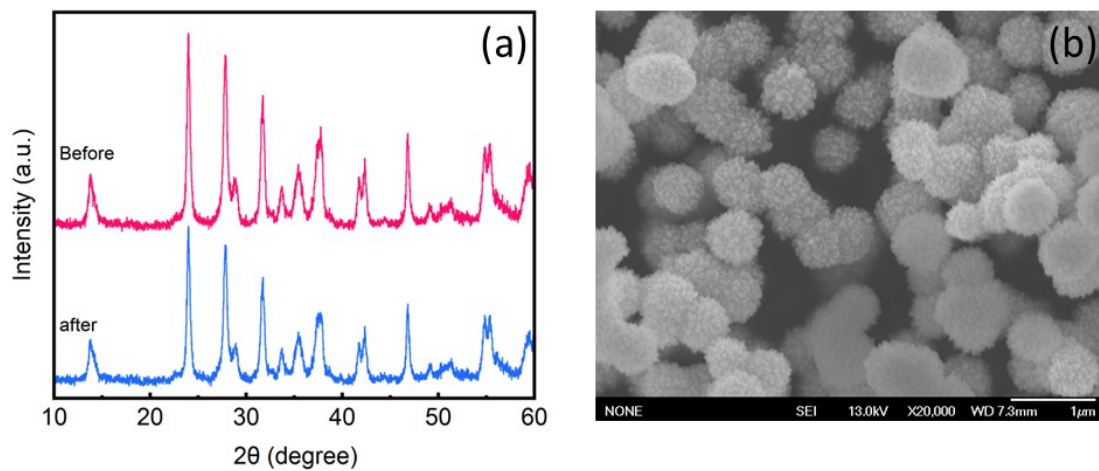
**Fig. S2.** XRD of as-prepared TOF samples with different reaction hours. (1h TOF1, 3h TOF3, 6h TOF6, 12h TOF12)



**Fig. S1.** SEM , TEM and HRTEM images of TOF (a, c and e) and TNF (b, d and f).



**Fig. S3.** Cyclic H<sub>2</sub> evolution curve for the TNF6 photocatalyst. The recycling experiment shows that the TNF6 sample does not exhibit any significant loss of activity, indicating that the catalysts are stable during photocatalytic H<sub>2</sub> production.



**Fig. S4.** (a) XRD patterns of the TNF6 nanoparticles before and after photocatalytic reaction. (b) SEM image of TNF6 nanoparticles after photocatalytic reaction. The XRD patterns of sample after photocatalytic reaction is the same as the one before reaction and the morphology is also not changed after photocatalytic reaction.