

## Supporting Information

Enhanced photoredox water splitting of Sb-N donor-acceptor pairs in TiO<sub>2</sub>

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## **Chemicals and materials**

Melamine, Tetrabutyl titanate (TBOT),  $\text{Sb}_2\text{O}_3$ , ammonium molybdate and chromium chloride hexahydrate were purchased from Sigma-Aldrich. Ammonia solution (28%), ethanol and triethanolamine (TEOA) were purchased from Sinopharm Chemical Reagent Co. (Shanghai, China).

## **Characterization**

X-ray diffraction (XRD) patterns were recorded on a Bruker D8 Advance Diffractometer (Germany) with Cu K $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ). X-ray photoelectron spectra (XPS) measurements were carried out using Thermo ESCALAB 250 instruments (USA) with non-monochromatic Al K $\alpha$  1486.6 radiation. Scanning electron microscope (SEM) was performed on a Hitachi S-3400N scanning electron microscope. The photoluminescence (PL) spectroscopy was measured using fluorescence spectrometer (Shimadzu RF-5301) at the excitation wavelength of 320 nm. The specific surface area was determined from the linear part of the BET equation ( $P/P_0 = 0.05\text{--}0.25$ ). The pore size distribution was derived from the desorption branch of the N<sub>2</sub> isotherm using the Barrett–Joyner–Halenda (BJH) method. Fourier transform infrared (FT-IR) spectra were measured using a Nicolet Magna-IR 750 spectrophotometer. UV-vis absorption spectra analysis was performed using a Shimadzu UV 3600 spectrometer. In photoelectrochemical measurements, MgSO<sub>4</sub> solution was used as electrolyte and the tests were performed by switching visual light ON/OFF with a duration of 30 s in a typical three-electrode cell.

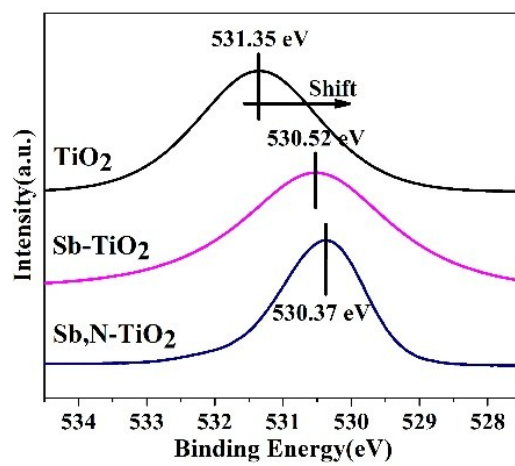


Fig. S1 The binding energy of O in TiO<sub>2</sub>, Sb-TiO<sub>2</sub> and Sb, N-TiO<sub>2</sub>.

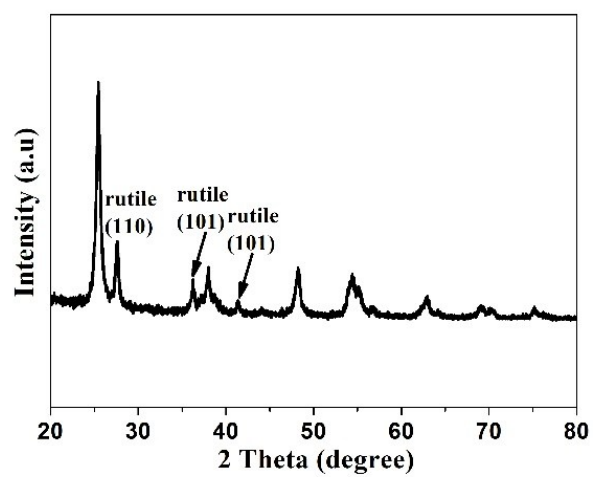


Fig. S2 XRD pattern of Sb<sub>3</sub>N-TiO<sub>2</sub> calcined at 550 °C.

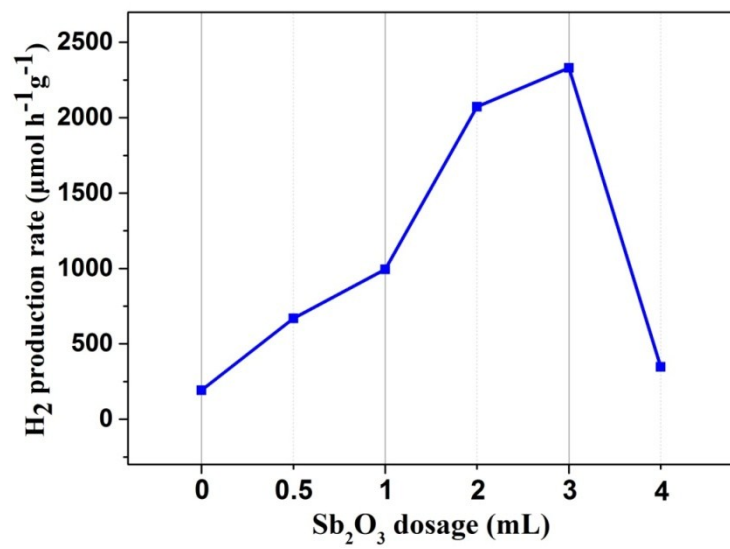


Fig. S3 Correlation between solar-light H<sub>2</sub> production and Sb<sub>2</sub>O<sub>3</sub> dosage.

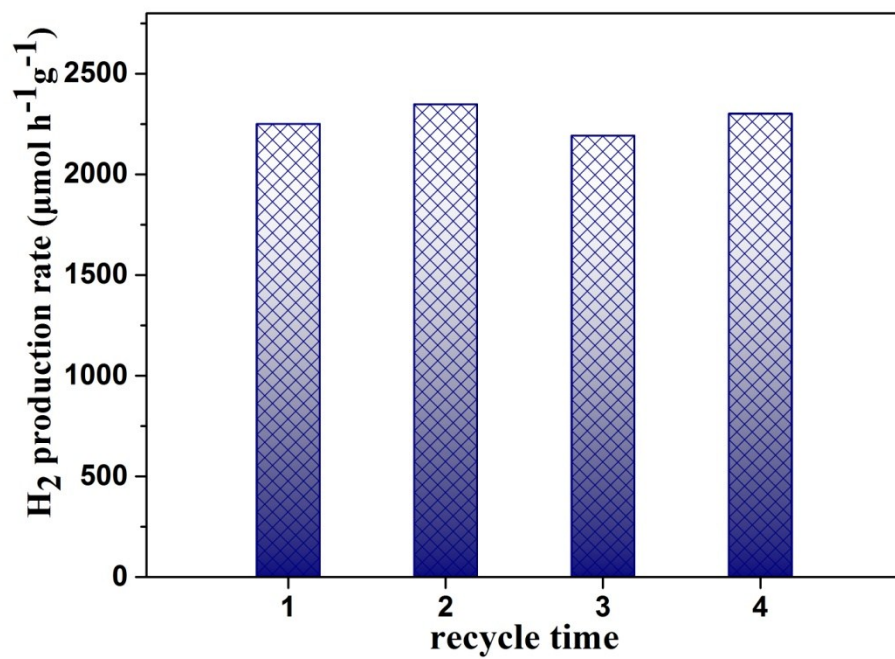


Fig. S4 Stability test of Sb<sub>3</sub>N-TiO<sub>2</sub> by adding 3 mL TEOA in the next cycle.

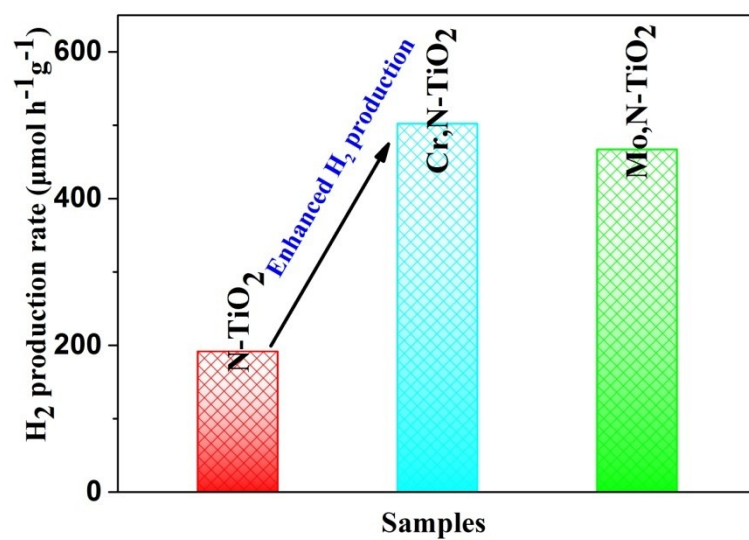


Fig. S5 Solar-light H<sub>2</sub> production of Cr,N-TiO<sub>2</sub> (synthesized in CrCl<sub>3</sub>·6H<sub>2</sub>O dosage of 0.3 g and melamine dosage of 3 mL) and Mo,N-TiO<sub>2</sub> (synthesized in H<sub>8</sub>MoN<sub>2</sub>O<sub>4</sub> dosage of 0.3 g and melamine dosage of 3 mL).



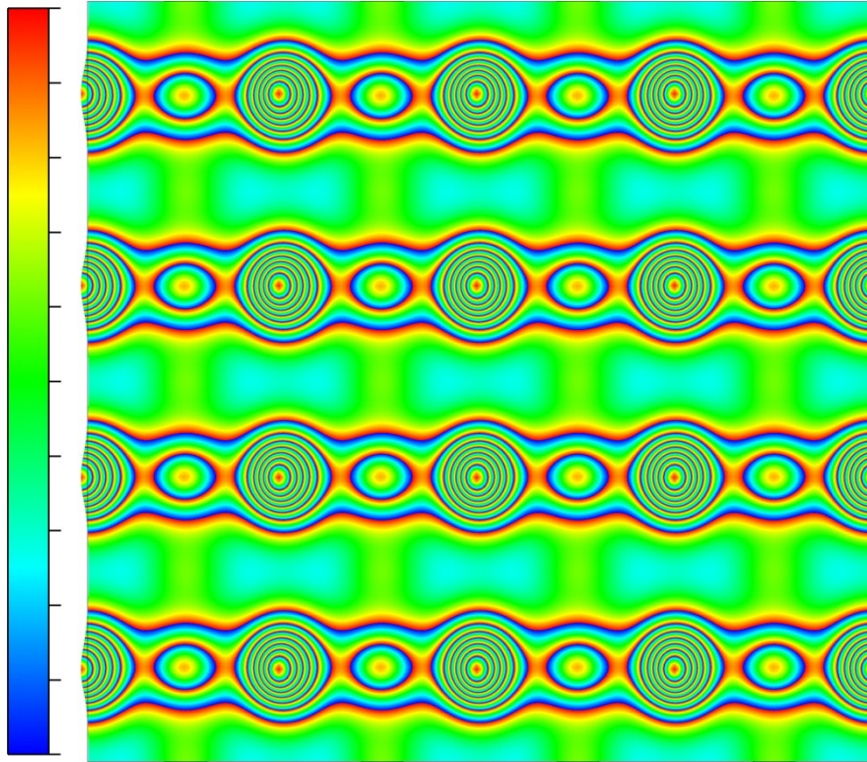


Fig. S6 Electron density in (001) plane of massive TiO<sub>2</sub>.