Supplementary Material (ESI) for Chemical Communications

Photocatalytic water oxidation on F, N co-doped TiO_2 with dominant exposed {001} facets under visible light

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1. Experimental details

Preparation of $TiOF_2$

The $TiOF_2$ cubes were synthesized according to a reported method.[1] In a typical synthesis, 5.0 ml of titanium butoxide ($Ti(OBu)_4$) was added dropwise to aqueous solution containing 10.0 ml of acetic acid (C_2H_5OOH) and 1.6 mL of 47% hydrofluoric acid solution under stirring conditions. The resulting gel was added to a Teflon-lined autoclave with a volume of 20 mL and kept at 473 K for 12 hours in an oven. After cooling down naturally to room temperature, the as-obtained products were collected and washed by water and ethanol for several times and dried at 373 K for 6 h.

Preparation of F and N co-doped anatase TiO_2 (TiOFN)

The as-prepared $TiOF_2$ cubes were heated at temperatures from 673 to 873 K for 1 h in NH_3 gas flow. The flow rate of NH_3 gas is 200 mL min^{-1}. After the nitridation treatment, the reaction is allowed to cool down to the room temperature in an NH_3 atmosphere. The as-obtained samples were further calcined at temperatures from 673 to 873 K for 1 h in air.

1.2 Characterization of photocatalysts

The synthesized products were comprehensively characterized by X-ray diffraction (XRD, Rigaku Miniflex), UV-Vis spectrometer (Shimadzu 2200), X-ray photoelectron spectroscopy (XPS, Thermo Escalab 250, a monochromatic Al KR x-ray source), scanning electron microscopy (JEOL 6300), and transmission electron microscopy (TEM, Philips Tecnei F20).

1.3 Photocatalytic reactions

The photocatalytic reactions were carried out in a quartz reaction cell connected to a closed gas circulation and evacuation system. 0.1 g of catalyst was suspended in 300 ml aqueous solution containing $AgNO_3$ (0.01 M) and La_2O_3 (0.2 g). The suspension was then thoroughly degassed and irradiated by a 300 W Xe lamp (Beijing Trusttech Co. Ltd., PLS-SXE-300UV) equipped with an optical cut-off filter ($\lambda > 420$ nm) to eliminate ultraviolet light and a water filter to remove infrared light. The temperature of the reactant solution was maintained at 293±5 K by a flow of cooling water during the reaction. The amount of O_2 produced was analyzed using an on-line gas chromatography.

References:

1. Xie, S.; Han, X.; Kuang, Q.; Fu, J.; Zhang, L.; Xie, Z.; Zheng, L. *Chem. Commun.*, *47*, 6722-6724.

2. Results of Characterizations

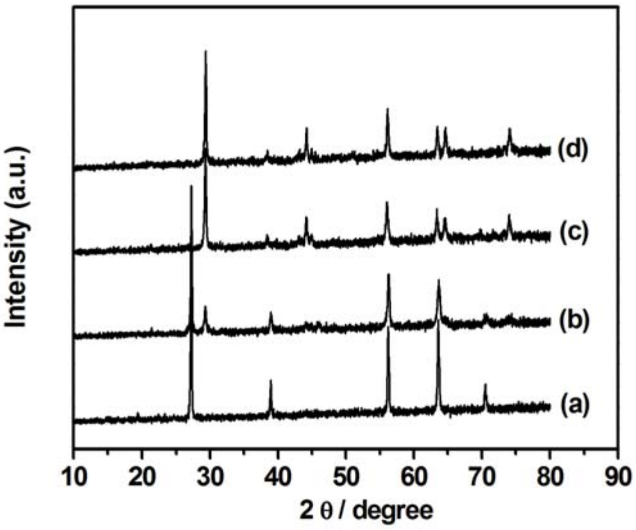

Fig. S1 X-ray diffraction patterns of (a) $TiOF_2$ precursor, and (b) TiOFN sample prepared by nitriding $TiOF_2$ in NH_3 gas flow at (b) 673, (c) 773, and (d) 873 K, respectively.

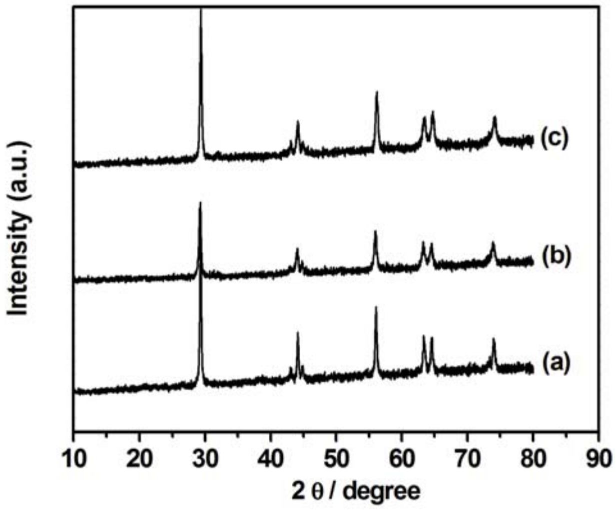

Fig. S2 X-ray diffraction patterns of samples obtained by calcining TiOFN samples in air at (b) 673, (c) 773, and (d) 873 K for 1 h, respectively.

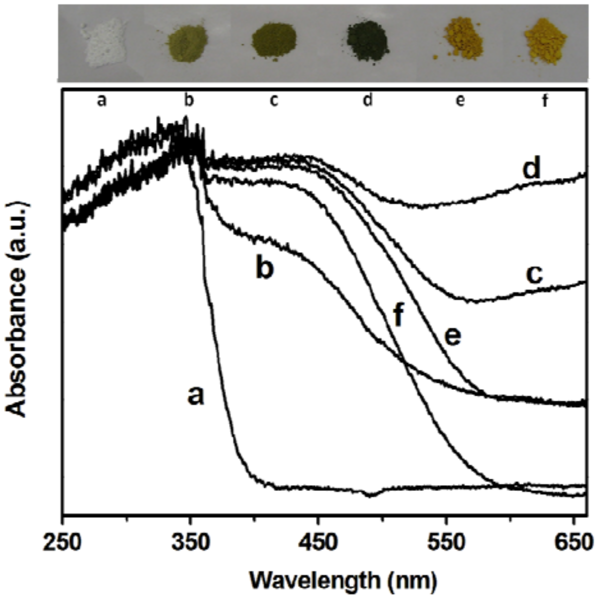

Fig. S3 Diffuse reflectance spectra of (a) $TiOF_2$ precursor, (b)-(d) TiOFN samples prepared by nitriding $TiOF_2$ in NH_3 gas flow at 673, 773, and 873 K, and (e)-(f) TiOFN sample obtained by calcining sample (c) in air at 773, and 873 K. The photos of the samples clearly show the change of the absorption properties of the resultant samples.

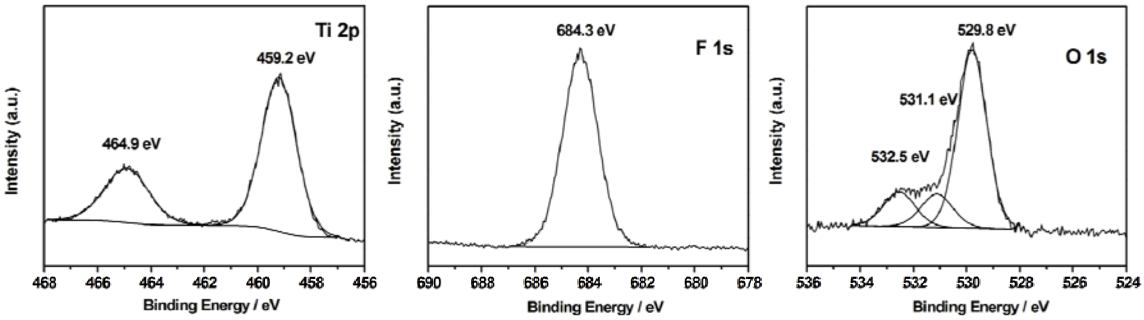

Fig. S4 High-resolution XPS spectra for Ti 2p, F 1s, and O1s measured on $TiOF_2$.

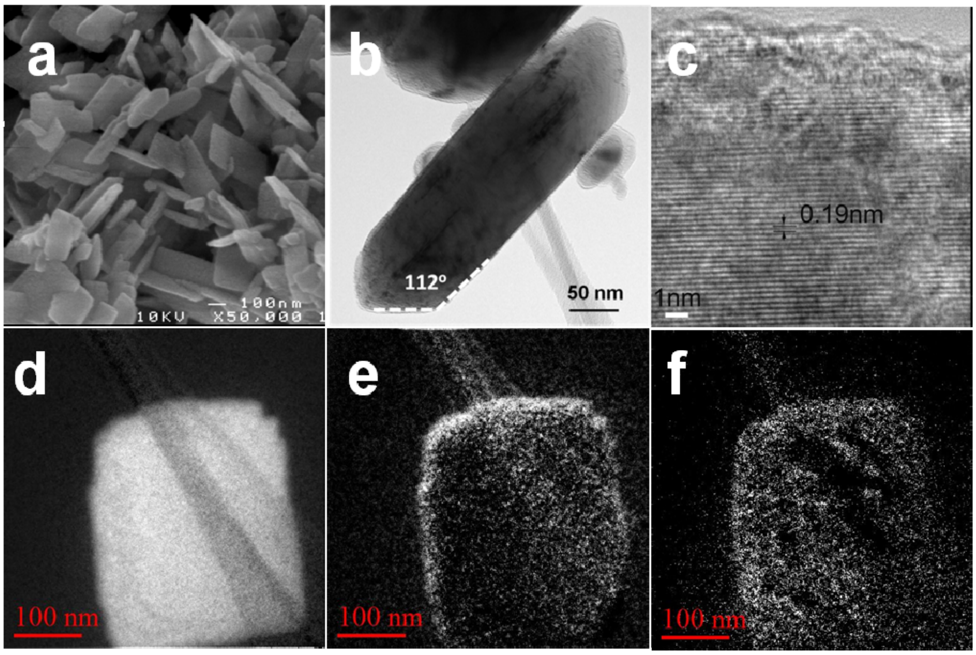

Fig. S5 SEM images of (a) TiOFN obtained by calcining as-prepared TiOFN sample in air at 773 K, (b) TEM image of a single erect platelet, and (c)-(f) HRTEM image, Ti, O, and F elemental maps for TiOFN sample.

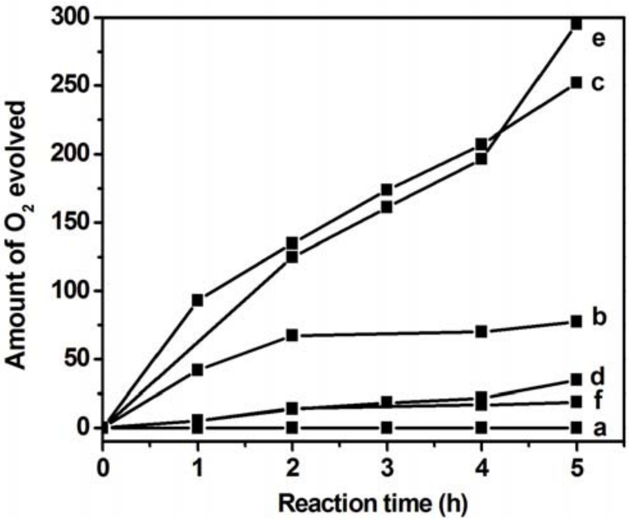

Fig. S6 Time courses of photocatalytic O_2 evolution on (a) TiF_2 precursor, (b)-(d) TiOFN prepared by calcining $TiOF_2$ precursor in ammonia gas flow at 673, 773, and 873 K, and (e)-(f) TiOFN obtained by calcining sample (c) in air at 673, and 773 K.