2D MXene-derived Nb_2O_5/C/Nb_2C/g-C_3N_4 heterojunctions for efficient nitrogen photofixation

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Experimental Section

Nb_2CT_x, Nb_2O_5/C/Nb_2C and Nb_2O_5 synthesis

Nb_2CT_x was prepared according to literature.[1] Nb_2O_5/C/Nb_2C was prepared according to literature.[2] Nb_2O_5 was prepared with the similar method as Nb_2O_5/C/Nb_2C except calcining the Nb_2CT_x at 850 °C in the CO_2 environment for three hours.[2]

Nb_2O_5/C/Nb_2C/g-C_3N_4 and Nb_2O_5/g-C_3N_4 heterojunctions

Nb_2O_5/C/Nb_2C/g-C_3N_4 heterojunctions were obtained by means of simple calcination of Nb_2O_5/C/Nb_2C and melamine mixture. Typically, a certain amount of melamine and Nb_2O_5/C/Nb_2C was added to 20 mL of distilled water and mixed by using ultrasound to obtain a homogeneous dispersion. Mixture was dried at 60 ^oC and ground to fine powder, which was calcined at 550 ^oC for 2 h in tube furnace at a heating rate of 5 ^oC min^{-1} under N_2. The

Nb_2O_5/C/Nb_2C/g-C_3N_4 1:1 with the Nb_2O_5/C/Nb_2C:melamine ratio 1:1 demonstrated the best catalytic performance. If no further notice provided, the Nb_2O_5/C/Nb_2C/g-C_3N_4 in this work alludes on Nb_2O_5/C/Nb_2C/g-C_3N_4 1:1. Nb_2O_5/g-C_3N_4 was prepared in the same way except Nb_2O_5 replaced Nb_2O_5/C/Nb_2C.

Characterization of the photocatalysts

Micromeritics ASAP 2020M automatic surface analyzer was used for BET analysis of the samples. Fourier transform infrared (FT-IR) spectras were obtained on the IRPrestige-21 instrument (400-2100 cm^{-1}). Scanning electron microscopy (SEM) was tested with the SU1510 (Hitachi, Japan). Transmission electron microscopy (TEM) measurements were tested with the FEI corporation Tecnai G2 F20. Powder X-ray diffraction (XRD) patterns were carried out with a LynxEye array detector/Bruker D8 Advance (5-80^o). X-ray photoelectron spectroscopy (XPS) spectra was conducted with Thermo Scientific Escalab 250Xi. The photocurrent performances were tested with an electrochemical system (CHI-660B, Chinehwa, Shanghai, China). The linear scanning curve was carried out with CHI-660B electrochemical workstation in Ar or N_2 saturated 0.5 mol L^{-1} Na_2SO_4 with a scan rate of 10 mV s^{-1}. The cyclic voltammetry was also carried out with CHI-660B electrochemical workstation, electrolyte: 5×10^{-3} M of $K_3Fe(CN)_6$ in 0.1 M KCl, scan rate: 50 mV s^{-1}. Photoluminescence (PL) spectra were tested with the Hitachi F-7000. UV–vis diffuse reflectance spectra (UV–vis DRS) were tested with a Hitachi UH4150 UV–vis DRS to investigate the optical properties of the samples (200-800 nm).

Photocatalytic N_2 fixation experiments

The photocatalytic N_2 fixation was carried out with a homemade 500 mL reaction cell with cooling system at 1atm pressure. 50 mg of photocatalyst was well suspended in 50 mL of double distilled water with 20 vol% CH_3OH. 99.999% N_2 with 100 mL min^{-1} was bubbled through the solution to achieve N_2 saturation in the dark for 1 hour. Reaction was carried out with the irradiation of Xe lamp around 0.5 W cm^{-2} with magnetic stirring. NH_4^+ concentration was analyzed with Nessler's reagent method.[3]

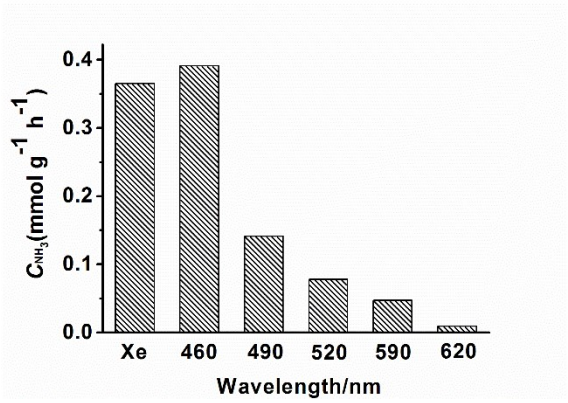

Fig. S1. Wavelength dependence of the nitrogen photofixation efficiency over Nb_2O_5/C/Nb_2C/g-C_3N_4.

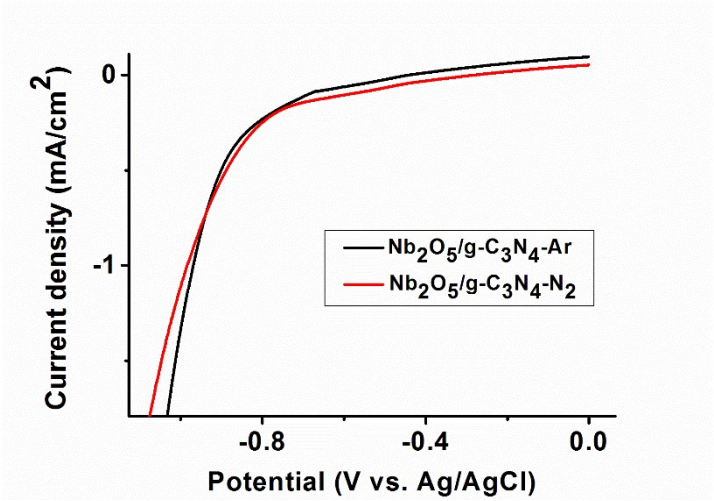

Fig. S2. Linear sweep voltammetry of Nb_2O_5/g-C_3N_4 under Ar or N_2 saturated solution.

References

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