2D MXene-derived Nb$_2$O$_5$/C/Nb$_2$C/g-C$_3$N$_4$ heterojunctions for efficient nitrogen photofixation

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Experimental Section

**Nb$_2$CT$_x$, Nb$_2$O$_5$/C/Nb$_2$C and Nb$_2$O$_5$ synthesis**

Nb$_2$CT$_x$ was prepared according to literature. Nb$_2$O$_5$/C/Nb$_2$C was prepared according to literature. Nb$_2$O$_5$ was prepared with the similar method as Nb$_2$O$_5$/C/Nb$_2$C except calcining the Nb$_2$CT$_x$ at 850 °C in the CO$_2$ environment for three hours.

**Nb$_2$O$_5$/C/Nb$_2$C/g-C$_3$N$_4$ and Nb$_2$O$_5$/g-C$_3$N$_4$ heterojunctions**

Nb$_2$O$_5$/C/Nb$_2$C/g-C$_3$N$_4$ heterojunctions were obtained by means of simple calcination of Nb$_2$O$_5$/C/Nb$_2$C and melamine mixture. Typically, a certain amount of melamine and Nb$_2$O$_5$/C/Nb$_2$C was added to 20 mL of distilled water and mixed by using ultrasound to obtain a homogeneous dispersion. Mixture was dried at 60 °C and ground to fine powder, which was calcined at 550 °C for 2 h in tube furnace at a heating rate of 5 °C min$^{-1}$ under N$_2$. The
Nb₂O₅/C/Nb₂C/g-C₃N₄ 1:1 with the Nb₂O₅/C/Nb₂C:melamine ratio 1:1 demonstrated the best catalytic performance. If no further notice provided, the Nb₂O₅/C/Nb₂C/g-C₃N₄ in this work alludes on Nb₂O₅/C/Nb₂C/g-C₃N₄ 1:1. Nb₂O₅/g-C₃N₄ was prepared in the same way except Nb₂O₅ replaced Nb₂O₅/C/Nb₂C.

Characterization of the photocatalysts
Micromeritics ASAP 2020M automatic surface analyzer was used for BET analysis of the samples. Fourier transform infrared (FT-IR) spectra 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°). 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 N₂ saturated 0.5 mol L⁻¹ Na₂SO₄ 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 K₃Fe(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 N₂ fixation experiments
The photocatalytic N₂ 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% CH₃OH. 99.999% N₂ with 100 mL min⁻¹ was bubbled through the solution to achieve N₂ 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. NH₄⁺ concentration was analyzed with Nessler's reagent method.³

![Fig. S1. Wavelength dependence of the nitrogen photofixation efficiency over Nb₂O₅/C/Nb₂C/g-C₃N₄.](image-url)
Fig. S2. Linear sweep voltammetry of Nb$_2$O$_5$/g-C$_3$N$_4$ under Ar or N$_2$ saturated solution.

References