Strengthening reactive metal-support interaction to stabilize Ni species on nitrogen vacancies of g-C_3N_4 for boosting photocatalytic H_2 production

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Experimental Details

Material Characterization

The X-ray diffraction (XRD) patterns were taken in reflection mode (Cu Kα radiation) on a Rigaku D/MAX-2500 diffractometer. The transmission electron microscopy (TEM) images were obtained by a scanning transmission electron microscope (JEOL 3010). X-ray photoelectron spectroscopy (XPS) spectra were recorded on AXIS-HSi spectroscope (Kratos Analytical) with a monochromated Al Kα X-ray source (1486.7 eV). The UV-vis diffuse reflectance spectra were recorded using a UV-vis

spectrophotometer and barium sulfate as the reflectance standard. Fourier transform infrared spectroscopy (FT-IR) were collected on Perkin-Elmer FTIR spectrophotometer (ThermoSmart-iTR) in the range of 4000-400 cm^{-1} using KBr technique. Photoluminescence spectra were performed using an Fluoromax-3 spectrometer (Horiba Scientific) with excitation wavelength of 350nm.

Photocatalytic H_2 production performance

The photocatalytic H_2 evolution was evaluated under visible light. All the photocatalytic measurements were executed in a closed gas evacuation and circulation catalytic reaction system equipped with a top-irradiation optical quartz window. Generally, 20 mg catalyst was suspended in a mixed solution of 90 ml distilled water and 10 ml triethanolamine (TEOA) by a magnetic stirrer. The suspension was purged with an argon flux for 30 min prior to irritation to remove the air and ensure the anaerobic condition. A 300 W Xe lamp was served as visible-light source with a filter ($\lambda > 420$ nm), and the temperature of the reaction systems was kept around the room temperature by a flowing of cooling during the test. During irradiation, the evolved H_2 gas was collected every hour and quantified by a gas chromatography using thermal conductivity detector (TCD).

Photoelectrochemical Measurement.

The photoelectrochemical tests were performed using an electrochemical working station (CHI760E) in 0.5 M Na_2SO_4 with Pt foil and Ag/AgCl electrode as counter and reference electrodes, respectively, within a three-electrode cell. The photocatalyst coated at ITO glass as a working electrode. A 300 W Xe lamp with a filter ($\lambda > 420$ nm) was applied as the light source.

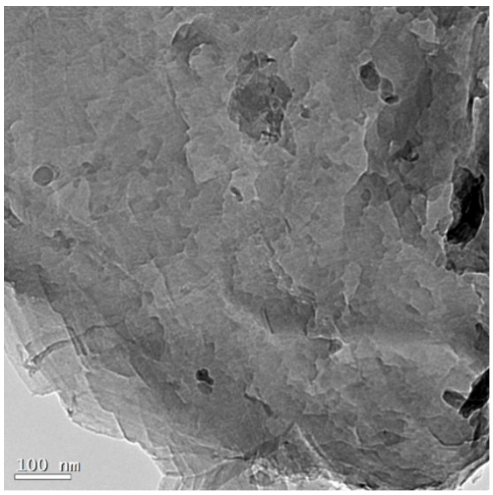

Fig.S1 TEM image of CN

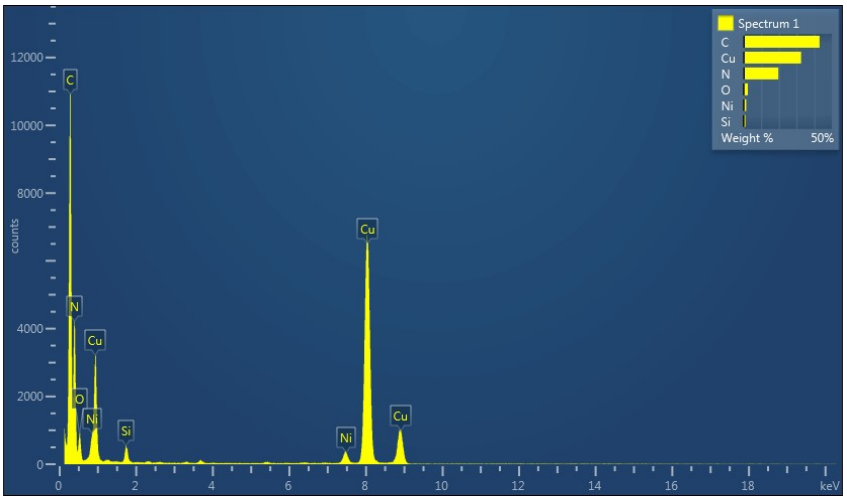

Fig.S2 EDS of DCN-Ni_2

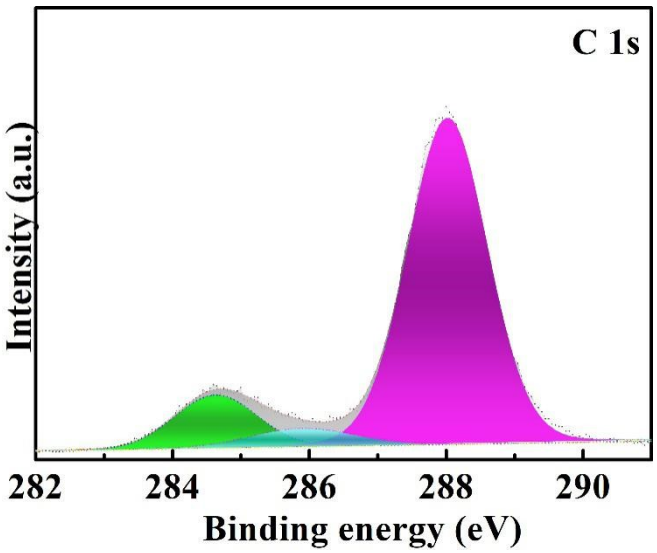

Fig. S3 XPS spectra of C 1 s over CN-Ni_2

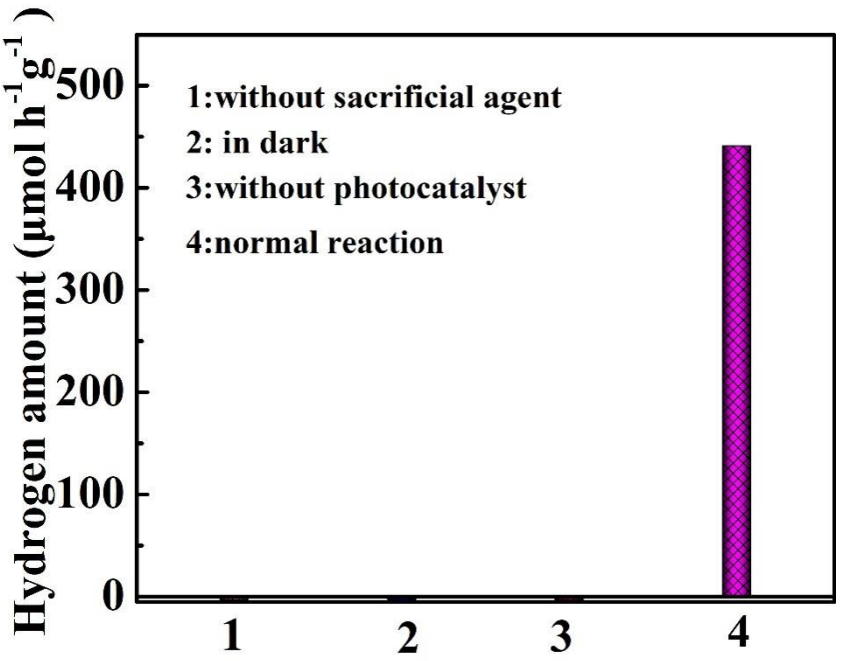

Fig. S4 The control experiments with different experiment condition

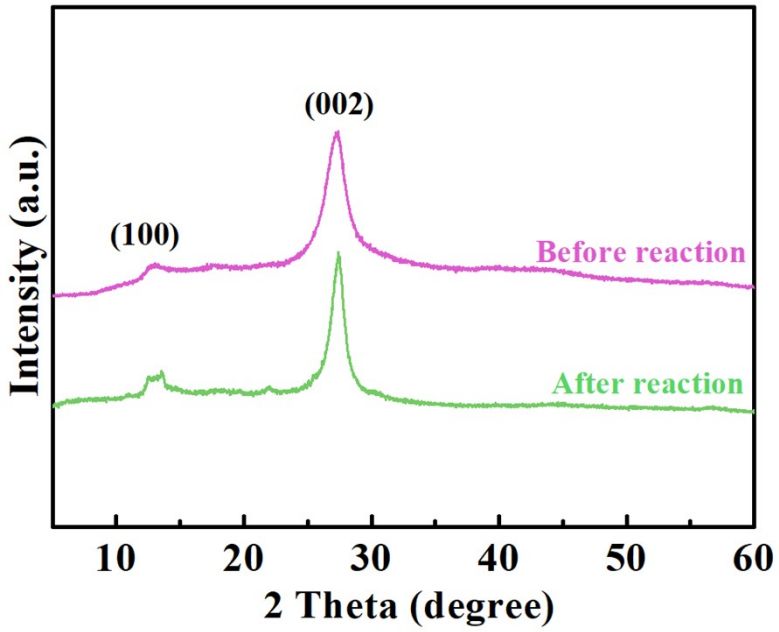

Fig S5. XRD patterns of DCN-Ni_2 before and after reaction.

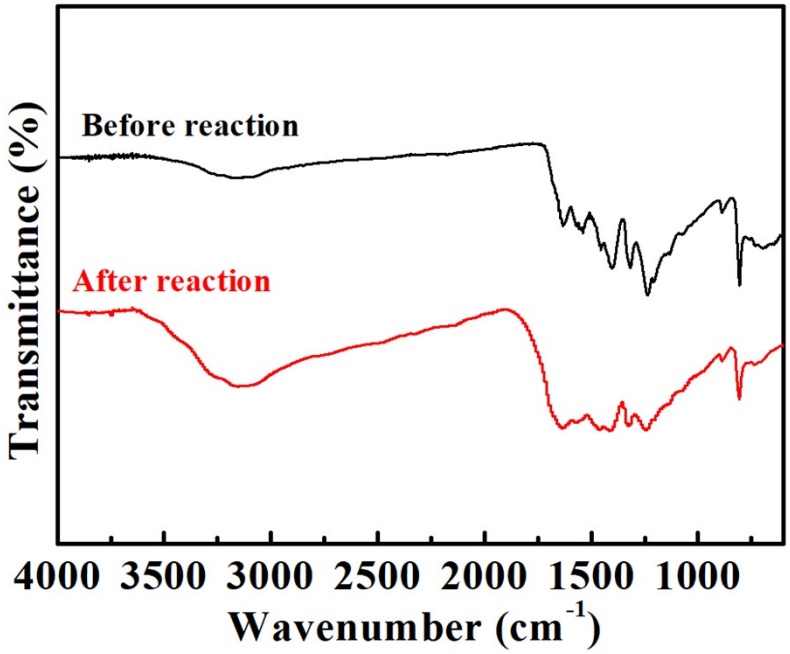

Fig S6. FT-IR of DCN-Ni_2 before and after reaction.

Table S1. The control experiments with different experiment condition

Sample	Sacrificial agent	Light	H_2 evolution
DCN-Ni_2	TEOA	xenon lamp	441 μmol $h^{-1}g^{-1}$
Without	TEOA	xenon lamp	0
DCN-Ni_2	TEOA	without	0
DCN-Ni_2	without	xenon lamp	0

Table S2. Compare with several reported catalyst used for hydrogen generation.

Sample	Efficiency of H_2 evolution	Ref.
DCN-Ni_2	441 μmol $h^{-1}g^{-1}$	This work
Ni@g-C_3N_4	168.2 μmol $h^{-1}g^{-1}$	1
Ni NPs on g-C_3N_4	440.8 μmol $h^{-1}g^{-1}$	2
FeP/g-C_3N_4	77.9 μmol $h^{-1}g^{-1}$	3
ZnO/Fe_2O_3/g-C_3N_4	250 μmol $h^{-1}g^{-1}$	4
CuO/g-C_3N_4	30.8μmol $h^{-1}g^{-1}$	5
In_2O_3@g-C_3N_4	258.8 μmol $h^{-1}g^{-1}$	6

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