

Supporting Information

Ni-Nanoparticles-Bound Boron Nitride Nanosheets

Prepared by Radiation-Induced Reduction-

Exfoliation Method and Their Catalytic Performance

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1) XPS spectra of h-BN treated in the mixture of isopropanol/water containing different metal ions (Ag⁺, Co²⁺, and Cu²⁺):

The h-BN powder (40 mg) was dispersed ultrasonically in 100 mL of an aqueous solution of isopropanol (IPA) (the relative volume of IPA and water ($V_{\text{IPA}}:V_{\text{water}}$) is 1:6) containing 0.1 mol/L of the corresponding metal salt (AgNO₃, CoCl₂, and CuCl₂). The dispersion was kept at 70 °C for over 48 h. In order to characterize the interaction between h-BN and metal ions, the solid particles could be separated by filtering and freeze-drying for XPS analysis. The XPS spectrum of different treated complex was

shown in Figure S1. The peaks at 374.25 eV and 368.3 eV in Ag 3d spectra were corresponding to Ag,¹ and the peaks in Co 2p spectra indicated the presence of Co(OH)₂.² In addition, the peaks in Cu 2p were assigned to CuCl₂.³⁻⁴ There was no new peak appearing, which indicated that there was little interaction between these ions and h-BN.

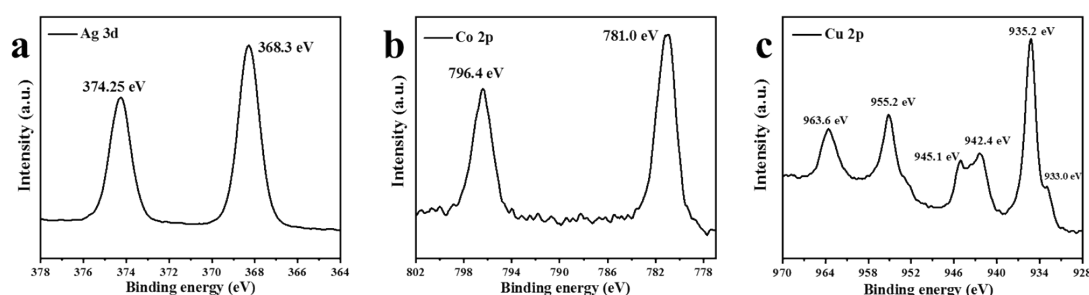


Figure S1. XPS spectra of h-BN treated in the mixture of isopropanol/water containing different metal ions: (a) Ag⁺, (b) Co²⁺, and (c) Cu²⁺.

2) XRD and TEM characterizations of the product after the addition of NaBH₄ into the dispersion of the Ni²⁺-intercalated h-BN

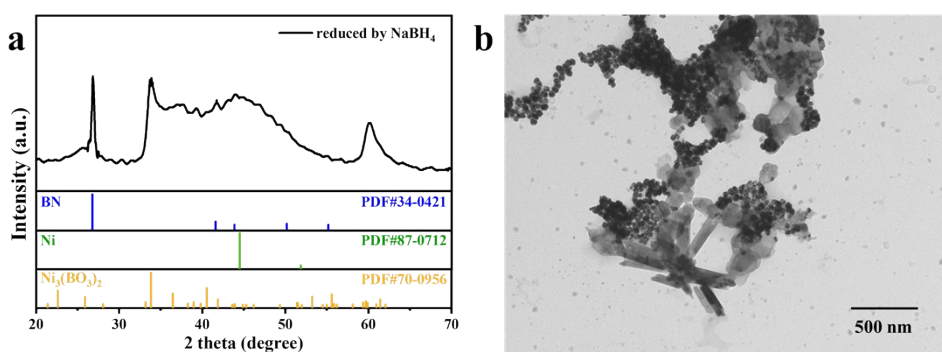


Figure S2. The XRD diagram (a) and TEM image (b) of the black precipitate produced after the addition of NaBH₄ into the dispersion of the Ni²⁺-intercalated h-BN

3) γ -Ray radiation on the mixture of isopropanol/water containing h-BN and CTAC:

h-BN powder (40 mg) was dispersed ultrasonically in 100 mL of the aqueous solution

of isopropanol (IPA) containing 12.5 mmol/L of hexadecyltrimethylammonium chloride (CTAC). The mixture was deoxygenated by bubbling nitrogen and sealed with a standard plug and then irradiated at a dose rate of 90.2 Gy/min and a total dose of 259.8 kGy at room temperature under continuous magnetic stirring. After the irradiation, solid product in the irradiated mixture was separated by centrifugation, and rinsed by deionized water to remove extra CTAC. The XRD spectrum and TEM image of the obtained powder were shown in Figure S2. Compared with the spectrum of pristine h-BN (Figure S2a), the crystalline structure of the irradiated powder is basically unchanged, and the morphology of the irradiated h-BN particles are still agglomerated particles (Figure S2b). No peeled thin sheets can be observed. It is convinced that surfactant molecules could not achieve the exfoliation preparation of BNNS alone in this system.

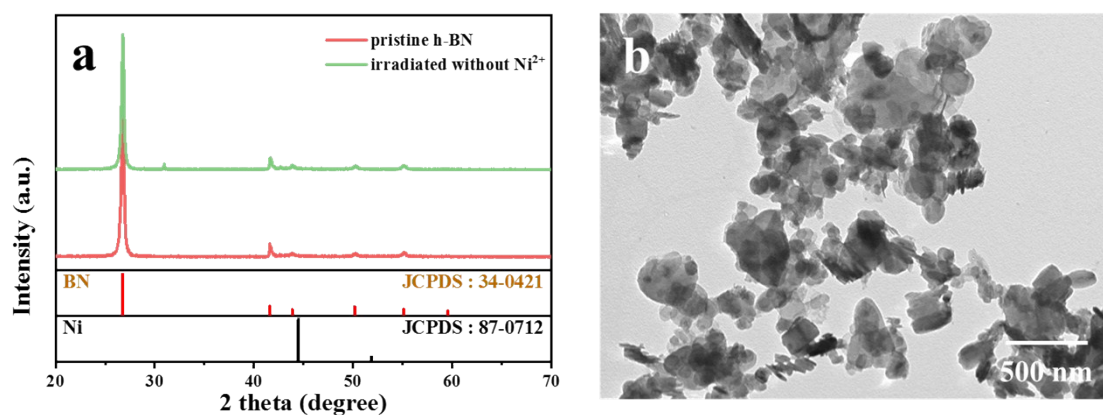


Figure S3. XRD spectra (a) and TEM image (b) of the h-BN particles dispersed in the mixture of IPA/water containing CTAC after being irradiated by γ -ray radiation.

4) Preparation of free NiNP by γ -Ray radiation

NiCl₂·6H₂O (2.376 g) was dissolved in 100 mL of the aqueous solution of isopropanol (IPA) containing 12.5 mmol/L of hexadecyltrimethylammonium chloride (CTAC). The

mixture was deoxygenated by bubbling nitrogen and sealed with a standard plug, and then irradiated at a dose rate of 90.2 Gy/min and a total dose of 259.8 kGy at room temperature under continuous magnetic stirring. After the irradiation, solid product in the irradiated mixture was separated by centrifugation, and rinsed by deionized water to remove extra CTAC. The TEM image of free NiNP and its corresponding were shown in Figure S3. The morphology of free NiNP was mainly spherical particles, and its size was mostly 16 ~ 22 nm according to statistics.

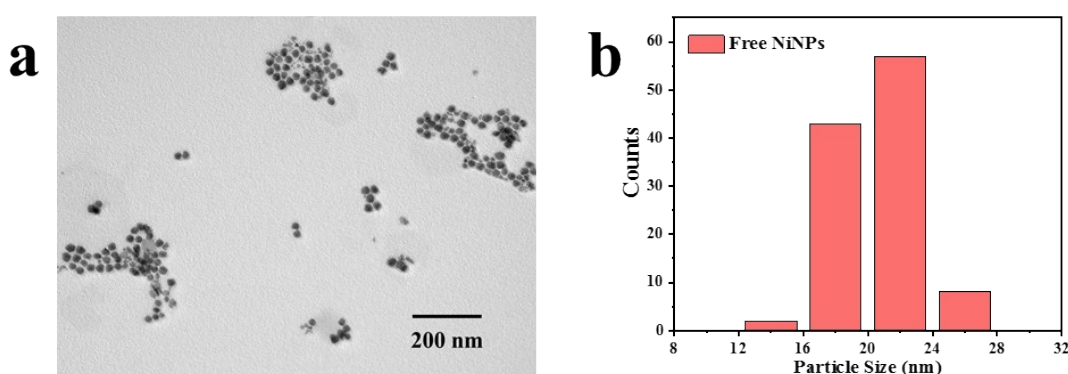


Figure S4. TEM image (a) and particles size histogram (b) of free NiNP (110 particles were measured).

5) The determination of the loading capacity of NiNP on Ni/BNNS

From the aqueous dispersion of the product, two pieces of samples were taken. One of them (20 mL) was dried directly, and the resulting solid was weighed for calculating the Ni/BNNS content in the sample (X mg/mL). Another (0.5 mL) was diluted with 0.1 M hydrochloric acid to 50 times the volume to let NiNP convert into Ni^{2+} ions, and then the system was filtrated through a 0.22 μm filter to separate the solid content. The filtrate solution was applied to the AES measurement. The calculated NiNP content in the original solution was Y mg/mL (ignoring the mass difference between nickel ions

and nickel atoms). Finally, the load W_{Ni} of NiNP in Ni/BNNS was calculated according to the following formula:

$$W_{Ni} = \frac{Y}{X} \times 100\% \quad (\text{Eq. S1})$$

References

- (1). Anthony, M. T.; Seah, M. P. XPS - Energy Calibration of Electron Spectrometers
.1. An Absolute, Traceable Energy Calibration and the Provision of Atomic Reference Line Energies. *Surf. Interface Anal.* **1984**, *6*, 95-106.
- (2). McIntyre, N. S.; Cook, M. G. X-Ray Photoelectron Studies on Some Oxides and Hydroxides of Cobalt, Nickel, and Copper. *Anal. Chem.* **1975**, *47*, 2208-2213.
- (3). Vasquez, R. P. CuCl₂ by XPS. *Surf. Sci. Spectra* **1993**, *2*, 160-164.
- (4). Okada, K.; Kotani, A. Multiplet Structures of Cu 2p-XPS in La₂CuO₄, CuO and Cu Halides. *J. Phys. Soc. Jpn.* **1989**, *58*, 2578-2585.