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Selected growth of second phase on BiOBr facets via spatial charge separation towards enhanced photocatalysis activity

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ABSTRACT

The recombination rate of electrons and holes determine the photocatalytic effect. Au nanoparticle (NPs) were loaded on the (110) plane of BiOBr via an ultrasonic method while PbO₂ NPs were loaded on the (001) plane. The ultrasound not only promoted the formation of the NPs but also resulted in directional transfer of electrons and holes in the different crystal planes, where electrons mainly exist in (110) planes while holes accumulate on (001) planes. Namely, ultrasonic waves caused BiOBr layer spacing to increase and destroy the balance of its built-in electric field. The loading of Au NPs enhanced the photocatalytic performance.

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Introduction

Recently, photocatalysts with enhanced performance have attracted much attention because of energy shortages and environmental pollution. Since Fujishima et al. [1] found that TiO₂ decomposed water in 1972, the application of semiconductor photocatalysts in solar energy conversion and photocatalytic degradation of organic pollutants has become a hot topic to explore. Due to the unique layered structure, suitable forbidden band width, high chemical stability and catalytic activity, BiOBr has a good response to visible light. However, electron and hole recombination rate in single semiconductor is high. The loading of noble metals is one of the important methods to reduce the recombination rate [2]. It is found that Au nanoparticles (NPs) are orientated on the (110) plane of BiOBr during ultrasound process. It is presumed that orientation migration of electrons and holes occurred during the ultrasound.

Electron–hole separation plays a key role in determining solar energy conversion efficiency of semiconductor-based materials such as electric catalytic [3–5], water splitting reaction [6–9] and photocatalysis [10–15]. After electron–hole pair separation, electrons and holes will be transferred to the surface of crystals [16–21]. In fact, many papers have reported on different crystal facets with equable engineering [22–27]. This phenomenon is ascribed to their

dependence of the energy levels [28]. This resulted in electrons and holes are transferred to different crystal facets [29]. For example, Li et al. [30] prepared BiVO₄ and deposited metal and metal oxide on the various facets under photo-irradiation to indicate the facet-selective phenomenon, which further draws the crystal heterogeneous junction theory [31]. Thus, the certain crystal facets of a semiconductor prefer reduction while others favour oxidation. Similarly, when the co-catalysts were directionally loaded on the right crystal facets, the sample will have high photocatalytic activity. Furthermore, in this paper, we proposed a completely new mechanism to explain the phenomenon of the directed migration of electrons and holes presented in BiOBr crystals during ultrasound. A traditional hydrothermal method [32,33] was used to prepare BiOBr as the model photocatalyst and study its space separation phenomenon via ultrasonic irradiation.

BiOBr/Au and BiOBr/PbO₂ samples were prepared, in which Au NPs were loaded on the (110) crystal facets of BiOBr and the PbO₂ NPs were deposited on the (001) crystal facets. The band gaps of PbO₂ NPs and BiOBr do not match and no improved performance observed. However, PbO₂ NPs just to assist to demonstrate the phenomenon of targeted migration of electrons and holes during ultrasound. The surface Plasmon resonance (SPR) effect and good conductivity of Au NPs enhance the photocatalytic performance of BiOBr. Loading different loci of metal and metallic oxide has shown a facet-selective phenomenon and clearly indicated the electrons and holes to various facets. The growth mechanism of BiOBr/Au and BiOBr/PbO₂ heterojunction was proposed by a series of characterization methods and photocatalytic experiments.

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Experimental

Materials

Bismuth nitrate pent hydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$), potassium bromide (KBr), chloroauric acid ($\text{HAuCl}_4 \cdot 4\text{H}_2\text{O}$), lead chloride (PbCl_2), anhydrous ethanol ($\text{C}_2\text{H}_6\text{O}$) were used for the preparation of samples. The pure water (resistivity of $18 \text{ M}\Omega/\text{cm}$) used during sample preparation was obtained from a Milli-Q synthesis system. All reagents are analytical grade and they were used directly without any further purification.

Synthesis of BiOBr crystals

Ultrasound was supplied by a KQ-500E ultrasonic cleaning instrument. Sample BiOBr was prepared via a hydrothermal method. 0.4851 g of $\text{Bi}(\text{NO}_3)_3 \cdot \text{H}_2\text{O}$ was dissolved in 30 ml of pure water with vigorous magnetic stirring for 2 h. 0.119 g of KBr was added to 20 ml of water. Then, a $\text{Bi}(\text{NO}_3)_3 \cdot \text{H}_2\text{O}$ solution was slow added to a KBr solution with vigorous stirring for 30 min. The mixture solution was transferred into a 100 ml Teflon-lined stainless autoclave and then hydrothermally treated at 160°C for 12 h. BiOBr samples were centrifuged, washed by water and anhydrous ethyl alcohol and dried at 60°C for 12 h.

Via ultrasonic irradiation to synthesis of BiOBr/ PbO_2 composite

PbO_2 NPs were grown on BiOBr samples by ultrasonic irradiation. The as-synthesized BiOBr powders (0.2 mmol) and 0.16 ml of KIO_4 (50 mM) were dispersed in 50 ml of water. KIO_4 was as an electron trapping agent. The solution was sonicated for 10 min, meanwhile, 0.08 ml of PbCl_2 (50 mM) was added slowly into the mixed solution. Then, a BiOBr/Au composite sample was obtained by centrifuging and drying. This sample is named as BiOBr/ PbO_2 (U).

Via ultrasonic irradiation to synthesis of BiOBr/Au composite

Au NPs were grown on BiOBr samples by ultrasonic irradiation. The as-synthesized BiOBr powders (0.2 mmol) were dispersed in 50 ml mixed solution (the volume ratio of water and ethyl alcohol is 4:6). Mixed solution was sonicated for 10 min, meanwhile, 0.08 ml of $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ (50 mM) was added slowly into the mixed solution. The interval of color was changed from white to purple. This sample is named as BiOBr/Au(U).

Synthesis of BiOBr/Au composite via photo-deposited

Au NPs were deposited on BiOBr samples by photo-deposited. As-synthesized BiOBr powders (0.2 mmol) were dispersed in 50 ml mixed solution (the volume ratio of water and anhydrous ethyl alcohol is 4:6). Mixed solution was irradiated by a 300 W Xe lamp ($\lambda \geq 420 \text{ nm}$) for

90 min, meanwhile, 0.08 ml of $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ (50 mM) was added slowly into the mixed solution. The interval of color changed from white to purple. This sample is named as BiOBr/Au(I).

Synthesis of BiOBr/ PbO_2 composite via photo-deposition

PbO_2 NPs were grown on BiOBr samples by photo-deposited. As-synthesized BiOBr powders (0.2 mmol) and 0.16 ml of KIO_4 (50 mM) were dispersed in 50 ml water. The solution was irradiated by a 300 W Xe lamp ($\lambda \geq 420 \text{ nm}$) for 90 min; meanwhile, 0.08 ml of PbCl_2 (50 mM) was added slowly into the mixed solution. This sample is named as BiOBr/ PbO_2 (I) (Table 1).

Characterization

The X-ray diffraction (XRD) patterns of as-synthesis samples were characterized via an X-ray diffract meter (Bruker D8, Germany) with a $\text{Cu K}\alpha$ radiation source and recorded in the 2θ range $10\text{--}80^\circ$. Morphology observation was carried out by scanning electron microscopy (SEM) (QUANTA 250 FEG, FEI, America) and transmission electron microscopy (TEM) (FEIG2F20). An UV/visible spectrophotometer (U-4100, Hitachi) are used to record the UV-visible diffuse reflectance and absorption spectra. The photoluminescence spectra were obtained on a Hitachi F-4600 Fluorescence spectrometer at room temperature using 400 nm as excitation wavelength. The total organic carbon analysis (TOC) was obtained on Shimadzu TOC-VCPN. A standard three-electrode cell was used to characterize photocurrent. Prepared 0.1 M Na_2SO_4 solution was as the electrolyte. Platinum wire was as the counter electrode, Ag/AgCl was as the reference electrode and an as-synthesis sample was as the working electrode.

Photocatalytic tests

As prepared BiOBr, BiOBr/Au and BiOBr/ PbO_2 were used to degrade rhodamine (RhB) under visible light. Samples (0.05 g) were added into an RhB solution (50 ml, 20 mg l^{-1}). In order to get the equilibrium absorption state, the mixed solution was stirred in the dark for 60 min. The 300 W Xe arc lamp was used to as the optical source and with a UV-cut off filter ($\lambda \geq 420 \text{ nm}$). Using a UV/visible spectrophotometer, the absorbance spectrum of RhB solutions was obtained. To observe the effect of the initial concentration and type of organic contaminants on the degradation, the initial concentration was reduced to half (50 ml, 10 mg l^{-1}) to degrade a methyl orange (MO) solution (50 ml, 10 mg l^{-1}).

Results and discussion

Description of different NPs grown on different facets of BiOBr

Fig. 1 shows the XRD pattern of BiOBr synthesized by a classical hydrothermal method. All diffraction peaks were matched to the

Table 1
Preparation conditions of samples.

Sample	Method	Au molar ratio (%)	PbO_2 molar ratio (%)	Solvent
BiOBr	N/A	N/A	N/A	N/A
BiOBr/Au(U_1)	Ultrasonic irradiation	5	N/A	H_2O
BiOBr/Au(U_2)	Ultrasonic irradiation	0.5	N/A	Ethanol and H_2O
BiOBr/Au(U_3)	Ultrasonic irradiation	1	N/A	Ethanol and H_2O
BiOBr/Au(U_4)	Ultrasonic irradiation	2	N/A	Ethanol and H_2O
BiOBr/ PbO_2 (U_1)	Ultrasonic irradiation	N/A	0.5	H_2O and KIO_4
BiOBr/ PbO_2 (U_2)	Ultrasonic irradiation	N/A	1	H_2O and KIO_4
BiOBr/ PbO_2 (U_3)	Ultrasonic irradiation	N/A	2	H_2O and KIO_4
BiOBr/Au (I_1)	Photo-deposition	2	N/A	Ethanol and H_2O
BiOBr/ PbO_2 (I_1)	Photo-deposition	N/A	2	H_2O and KIO_4

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