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Article

Fabrication of $\text{TiO}_2(\text{B})$ /anatase heterophase junctions in nanowires via a surface-preferred phase transformation process for enhanced photocatalytic activity

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ABSTRACT

Heterojunction fabrication is one of the most effective strategies for enhancing the photocatalytic performance of semiconductor photocatalysts. Here, $\text{TiO}_2(\text{B})$ /anatase nanowires with interfacial heterostructures were prepared through a three-step synthesis method, including hydrothermal treatment, H^+ exchange, and annealing. The phase structures of the nanowires in the bulk and on the surface during the annealing process were monitored by XRD and UV-Raman spectroscopy, respectively. SEM and TEM results indicate that the $\text{TiO}_2(\text{B})$ nanowires partially collapse and transform into anatase during the annealing process and the heterophase junction structure is formed simultaneously. On the basis of the phase structure together with morphology data, a phase-transformation mechanism was proposed. Photocatalytic activity was evaluated by hydrogen production and pollutant-degradation assays. The optimized structure of the photocatalyst contains 24% $\text{TiO}_2(\text{B})$ in the bulk and 100% anatase on the surface. The charge-carrier behavior during the photocatalytic process was investigated by photocurrent, electrochemical impedance spectroscopy (EIS), and photoluminescence (PL) spectroscopy, which revealed that the heterophase-junction structure in the bulk was responsible for the highly efficient charge separation and transportation, etc.; the anatase on the surface took control of the high surface-reaction activity.

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1. Introduction

Semiconductor photocatalysis has received extensive attention as a potential solution to the worldwide energy shortage and for environmental purification [1,2]. Since the photocatalytic water splitting reaction was first found by Japanese scientists Fujishima et al. in 1972 [3], the application of TiO_2 photocatalysis technology has been widely investigated [4–6]. TiO_2 is widely accepted as an environmentally benign material with

low toxicity, low cost, and high chemical stability. Over the last few decades, great effort has been devoted to developing highly efficient TiO_2 photocatalysts [7–9], not only due to its potential applications in photocatalysis, but also as model catalysts for understanding the mechanism of the photocatalysis process.

TiO_2 has various polymorphs, including anatase, rutile, $\text{TiO}_2(\text{B})$, and brookite. The photocatalytic activity of TiO_2 mainly depends upon its crystal structure. Generally, the anatase phase exhibits the best photocatalytic activity among the four

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TiO₂ polymorphs. However, the charge-separation efficiency of a single-phase semiconductor is obviously far from satisfactory. Therefore, many researchers have focused on how to effectively separate the photogenerated electron-hole pairs. Yu et al. [10–12] fabricated heterogeneous junctions between different photocatalysts with effectively enhanced activity. Zhang and coworkers [13] first revealed that the heterophase junction formed between anatase and rutile was the crucial factor responsible for the high charge-separation efficiency in mixed phase photocatalysts. Furthermore, the activity of the benchmark photocatalyst (P25 Degussa) can be significantly improved by tuning its heterophase junctions [14,15]. In addition, TiO₂-based heterophase junction structures, such as TiO₂(B)/anatase [16–18], anatase/rutile [19,20], anatase/brookite [21–23], brookite/rutile [24–26], TiO₂(B)/anatase/rutile [27,28], and anatase/rutile/brookite [29,30] have been studied. These heterophase junctions are type II heterojunctions, in which photogenerated electrons migrate from semiconductor A, with the higher conduction-band (CB) and valence-band (VB) levels, to semiconductor B with lower CB and VB levels upon irradiation with light, resulting in the spatial separation of electron-hole pairs [31].

TiO₂(B), as a minor phase, was first synthesized in 1980 by Marchand et al. [32], and has been widely used in lithium-ion batteries for its open lattice channels. TiO₂(B) exhibits enhanced power capabilities and bulk capacities compared to other TiO₂ polymorphs. This superiority is strongly linked to the structural features of this polymorph. The most distinct feature of TiO₂(B) is its reduced crystallographic density [33]. Although the photocatalytic activity of TiO₂(B) is lower than that of anatase, a well matched heterophase junction can be formed at the interface between these two phases, leading to an improvement in the photocatalytic activity for CO₂ reduction, hydrogen production, and pollutant degradation [34–36]. Generally, heterophase junctions between different phase structures can be formed by post-deposition or *in-situ* phase-transformation processes [37,38]. The phase transformation process is actually an atomic rearrangement process at the crystal interface. Thus, the two phases are naturally combined with each other in this case, which is more favorable for charge diffusion. Therefore, the mechanism of the phase-transformation process from TiO₂(B) to anatase is quite important for the construction of the heterophase junction. Unfortunately, only a few reports [39,40] have mentioned the phase-transformation process and the mechanism is still unclear.

Herein, TiO₂(B)/anatase nanowires with heterophase structures were prepared through a three-step synthesis method, including hydrothermal treatment, H⁺ exchange, and annealing. The phase structures of the nanowires in the bulk and on the surface during anneal were monitored by X-ray diffraction (XRD) and UV-Raman spectroscopy, respectively. This work first revealed the surface-preferred phase-transformation process of TiO₂(B) nanowires into anatase, and then constructed a highly efficient photocatalyst for both H₂ production and pollutant degradation.

2. Experimental

2.1. Chemicals

Titanium (IV) *n*-butoxide (C₁₆H₃₆O₄Ti, 99%) was purchased from J&K Chemical Co., Ltd (Beijing, China). Acetic acid (C₂H₄O₂, ≥99.5%), ethylene glycol (C₂H₆O₂, ≥99.0%), ethanol (C₂H₆O, ≥99.7%), sodium hydroxide (NaOH, ≥96.0%), hydrochloric acid (HCl, 36.0–38.0%), and hexachloroplatinic (IV) acid (H₂PtCl₆·6H₂O, ≥37.0%) were purchased from the Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). Rhodamine B (RhB) (≥95%) was obtained from Sigma-Aldrich (USA). All chemicals reagents were of analytical grade and used as received without any further purification. Deionized water was used in all experiments.

2.2. Catalyst preparation

Sodium titanate nanowires were first synthesized *via* hydrothermal treatment. Titanium (IV) *n*-butoxide (6 mL), used as the titanium source, and acetic acid (3 mL) were added to ethylene glycol (20 mL) and stirred for 15 min. NaOH solution (30 mL, 15 mol/L) was then added and stirred for 10 min. Afterwards, the suspension was transferred into a 100-mL Teflon-lined autoclave and maintained at 180 °C for 24 h. After the autoclave was cooled to room temperature, the sodium titanate nanowires were collected by centrifugation and washed with deionized water. Secondly, the obtained sodium titanate nanowires were transformed into hydrogen titanate nanowires by H⁺ exchange in 0.1 mol/L HCl solution for 12 h. Then, the sample was washed with water until the filtrate became neutral. The hydrogen titanate nanowires were obtained after drying at 80 °C for 12 h. Finally, to convert the hydrogen titanate into different phases, e.g., TiO₂(B), the mixed-phase TiO₂, or anatase, the hydrogen titanate nanowires were annealed in a muffle at different temperatures for 2 h. The as-prepared catalysts were denoted as 'HT-*T*', where HT is hydrogen titanate and *T* is the annealing temperature. The HT-*T* series of catalysts were used for subsequent characterization and photocatalytic reactions.

2.3. Material characterization

XRD patterns were obtained using a Rigaku MiniFlex 600 (Japan) diffractometer with Cu K_α radiation (V = 40 kV, I = 15 mA) over the 10°–70° (2θ) range and with a 0.02° step size; the patterns were normalized against the highest peak in each pattern. UV-Raman spectra were recorded on a Renishaw Raman spectrometer (Via + Reflex) system with a 325-nm excitation source. X-ray photoelectron spectroscopy (XPS) measurements were carried out using an Axis Ultra DLD XPS instrument (Kratos Analytical Ltd.) with an Al K_α radiator, and analyzed using casaXPS. Binding energies for the high-resolution spectra were calibrated by setting C 1s to 284.6 eV. UV-Vis diffuse reflection spectra were recorded on a UV-3600 spectrophotometer (Shimadzu, Japan) in the 200–800 nm range. The surface morphologies of the photocatalysts were observed by field emission scanning electron microscopy (FESEM, SU 8020, Hitachi

High-Technologies, Tokyo, Japan) operating at 5 kV. The SEM samples were prepared as follows: each sample was dispersed on a silicon slice, which was then fixed on a metal flat pallet by conductive adhesive tape; the sample was then treated with spray-gold for 80 s. Transmission electron microscopy (TEM) was performed on a JEM-2100 instrument (JEOL, Japan) under an acceleration voltage of 200 kV. Specific surface areas were obtained by acquiring N_2 -adsorption isotherms at a relative pressure of 10^{-6} MPa at 77 K on a Micromeritics ASAP2020 HD88 instrument. All photocatalysts were degassed at 300 °C for 4 h before adsorption experiments. Photoluminescence (PL) spectra were recorded on a Hitachi F-4600 fluorescence spectrophotometer with both excitation and emission slits set at 10 nm.

Photocurrent and electrochemical impedance spectroscopy (EIS) measurements were performed on a CorrTest CS310 electrochemical workstation (Wuhan CorrTest Instrument Co. Ltd., China) in a three-electrode cell with 1.0 mol/L Na_2SO_4 electrolyte at room temperature. The catalyst-loaded thin film, platinum sheet, and saturated Ag/AgCl electrode were used as working electrode, counter electrode, and reference electrode, respectively. The working electrodes were prepared on a 1 cm × 0.5 cm FTO glass substrate by the electrophoresis method followed by drying at 60 °C and annealing at 400 °C for 2 h in air. Photocurrent measurements (*i*-*t* curves) were carried out at 0.3 V (vs. saturated Ag/AgCl) under chopped illumination with a 300-W Xe lamp as the light source. The EIS data were acquired in the 0.01 Hz to 100 kHz frequency range at 0.3 V (vs. saturated Ag/AgCl).

2.4. Photocatalytic activity assays

2.4.1. Photocatalytic hydrogen production

Photocatalytic hydrogen evolution from water was carried out in a CEL-SPH2N photocatalytic activity evaluation system (Beijing China Education Au-Light Co., Ltd) at 15 °C, irradiated by a 300-W Xe lamp (PLS-SXE300C, Beijing China Perfectlight Co., Ltd). Typically, 50 mg of the photocatalyst was dispersed in an aqueous 100 mL solution containing a certain amount of sacrificial agent, followed by addition of 0.1 wt% Pt as the co-catalyst (*in-situ* photo-precipitation). To ensure that the reaction system was under inert condition, the suspension was degassed with a mechanical pump for 10 min to completely remove dissolved oxygen before illumination. The amount of hydrogen evolved was determined by gas chromatography (GC) using a thermal conductivity detector (TCD) with argon as the carrier gas.

2.4.2. Photocatalytic degradation of RhB

In a typical experiment, 50 mg of as-prepared photocatalyst was added to 150 mL of an aqueous RhB solution (4 mg/L) in a quartz photoreactor. The photocatalytic activity was evaluated under irradiation by a 300-W Xe lamp (CEL-HXF 300, Beijing China Education Au-Light Co., Ltd). Before irradiation, the mixture was stirred in the dark for 0.5 h to ensure adsorption equilibrium and the formation of a homogeneous suspension. The temperature of the reaction system was maintained at 15 ± 0.5

°C. During the reaction process, 2 mL of the reaction solution was taken out every 10 min and diluted to the same volume after the suspended photocatalyst was filtered off with a Millipore filter (pore size 0.22 μm). Then, the solution was analyzed on a TU-1901 UV-Vis spectrophotometer (Beijing Purkinje General Instrument Co., Ltd.) in the 400–700 nm range to monitor the reaction process.

3. Results and discussion

3.1. Structure and phase-transformation mechanism

3.1.1. Phase structure

The structures of hydrogen titanate annealed at different temperatures were investigated by XRD and UV-Raman spectroscopy. As shown in Fig. 1(a), when compared to the standard XRD patterns of $TiO_2(B)$ (JCPDS 46-1238) and anatase (JCPDF 21-1272), the sample gradually transformed from $TiO_2(B)$ into anatase as the annealing temperature was increased from 400 to 700 °C. The XRD patterns of HT-525, HT-575, HT-600, HT-610, and HT-650 can be assigned to the monoclinic phase of $TiO_2(B)$, and partly to the anatase phase (Fig. 1a). As the annealing temperature was increased, the intensities of the characteristic anatase peaks became stronger while the intensity of $TiO_2(B)$ decreased (Fig. 1(a)). These results indicate that $TiO_2(B)$ /anatase interfacial heterostructures are constructed by consecutive partial phase-transformation processes. The UV-Raman spectra of hydrogen titanate at different annealing temperatures are shown in Fig. 1(b). The peaks at 250 and 400 cm^{-1} are the typical characteristic Raman bands of $TiO_2(B)$ and anatase, respectively. As the annealing temperature was increased from 400 to 700 °C, the intensity of the Raman band at 250 cm^{-1} gradually decreased and nearly disappeared at 610 °C, while the intensity of the Raman band at 400 cm^{-1} was enhanced (Fig. 1(b)). The UV-Raman spectra for the HT-*T* series of photocatalysts indicate that the phase structure of $TiO_2(B)$ is totally transformed into the anatase phase in the temperature range between 610 and 700 °C. The phase transformation from $TiO_2(B)$ into anatase is definitely temperature dependent. However, the XRD patterns and UV-Raman spectra appear to give unsynchronized results because XRD is sensitive for the characterization of the bulk structure, while UV-Raman spectroscopy is a powerful tool for characterizing the surface structures of materials that absorb UV light [41,42].

3.1.2. BET surface area

The specific surface area of the photocatalyst is considered to be a vital factor that influences the photocatalytic activity. In most cases, a larger surface area provides more adsorption and/or reactivate sites for photocatalysis, which renders faster migration of charge carriers, resulting in the improvement of photocatalytic reaction rate. Fig. S1 shows the N_2 adsorption-desorption isotherms of the HT-400 to HT-700 photocatalysts. All isotherms are type II according to the Brunauer-Emmett-Deming-Teller (BDDT) classification, indicating that there are no porous structures in the photocatalysts. The spe-

Fig. 1. XRD patterns (a) and UV-Raman spectra (b) of the photocatalysts.

cific surface areas of the HT-*T* series of photocatalysts were calculated by BET multiplication. As shown in Fig. S1, the surface areas of the HT-*T* series of photocatalysts decrease with increasing annealing temperature. The area decreased from 32 m²/g for HT-400 to 7 m²/g for HT-700, indicating that the catalyst become more compact at higher temperatures.

3.1.3. Morphology

The morphologies of the HT-*T* series of photocatalysts were examined by SEM. In Fig. 2(a), the HT-400 photocatalyst shows a fibrous morphology and the average diameter of the HT-400 nanowires is approximately 230 nm. With increasing annealing temperature, the average diameter of the TiO₂ nanowires gradually decreased to around 85 nm (Fig. 2(a)–(g)). Comparing the SEM images of the HT-400 and HT-700 nanowires, the HT-700 nanowires have smaller diameters and smoother surface morphologies (Fig. 2(a) vs. Fig. 2(g)). As evidenced by XRD and UV-Raman spectroscopy, the HT-400 nanowires have TiO₂(B) phase structures, while the HT-700 nanowires have the anatase phase structure. Based on the SEM images, the structure of the TiO₂(B) skeleton partially collapses with increasing annealing temperature (Fig. 2(b)), and the anatase phase forms in the collapsed region. Further increases in temperature splits the TiO₂(B) by its collapsed regions (Fig. 2(c)–(e)) to finally form short rod-like anatase-phase struc-

tures (Fig. 2(f) and 2(g)). Apparently, large amounts of heterophase structures composed of original TiO₂(B) and newly formed anatase nanorods form during the phase-transformation process. The fiber-like morphology of the material was maintained during the phase-transformation process, and only bundles of TiO₂ nanorods were split into thinner anatase rods. As discussed above, when comparing the HT-400 and HT-700 nanowires, the HT-700 nanowires have the smaller sizes but still have small surface areas, which is possibly due to the TiO₂(B) phases behaving as open-channel structures, while the anatase phase has a compact structure. It is very interesting that the TiO₂(B) nanowires split rather than aggregate at high temperatures, as observed for other materials.

It should be mentioned that heterophase structures can form on both the surface of the catalyst and in the bulk. To better observe the detailed structure of the heterophase junction, HT-600, with more surface heterophase structures, was chosen for further TEM characterization. As shown in Fig. 3(a), the two typical TiO₂ phase structures were observed for the HT-600 nanowires, which were composed of 42% TiO₂(B) in the bulk and 17% TiO₂(B) on the surface. A heterophase structure (indicated by the black circle in Fig. 3(a)) was further demonstrated by high-resolution TEM (HRTEM), as shown in Fig. 3(b); the dark region shows the TiO₂(B) phase with lattice-spacing val-

Fig. 2. SEM images of the photocatalysts (a) HT-400, (b) HT-525, (c) HT-575, (d) HT-600, (e) HT-610, (f) HT-650, (g) HT-700.

Fig. 3. TEM (a) and HRTEM (b) images for the HT-600 photocatalyst. A: anatase, B: $\text{TiO}_2(\text{B})$.

ues of 5.05 and 3.53 Å corresponding to the (201) and (110) planes (JCPDS48-1238), respectively. The bright region is assigned to the anatase phase with typical lattice-spacing values of 3.52 and 1.88 Å corresponding to the (101) and (200) planes (JCPDS 21-1272), respectively. These results clearly show that a large amount of $\text{TiO}_2(\text{B})$ /anatase-heterophase junctions are formed in the nanowire structure.

3.1.4. Phase-transformation mechanism

Based on the standard XRD patterns (Fig. S2) and UV-Raman spectra (Fig. S3), the ratios of $\text{TiO}_2(\text{B})$ and anatase in the bulk structure and on the surface structure of the HT-*T* series of photocatalysts were calculated. As shown in Table 1, as the temperature is increased from 400 to 700 °C, the ratio of $\text{TiO}_2(\text{B})$ in the bulk structure of the photocatalyst gradually decreases from 100% to 0%. However, on the surface region, it quickly decreases to 0% at 610 °C. The above results show the unsynchronized $\text{TiO}_2(\text{B})$ -to-anatase phase-transformation process in the bulk and on the surface, and reveal that this phase transformation is a surface-preferred process. Combined with the SEM observations, the faster phase-transformation process on the surface region may due to the partial skeletal collapse of $\text{TiO}_2(\text{B})$. The collapsed region of the sample transforms into anatase and splits the material at the same time. This region will actually become the new surface of the material, thus increasing the content of anatase on the surface. Based on the above results, a model of for the temperature-dependent phase transformation process was proposed (Scheme 1), where the rate of phase transformation in the surface region (Scheme 1(a)) is faster than that in the bulk region (Scheme 1(b)). Taken together, the $\text{TiO}_2(\text{B})$ /anatase-interfacial hetero-

Scheme 1. Schematic diagram of the phase transformation on the surface (a) and in the bulk (b).

structures in nanowires possess higher anatase contents on the surface than in the bulk.

3.2. Effect of phase composition on photocatalytic performance

3.2.1. Photocatalytic hydrogen production

The photocatalytic performance of the HT-*T* series of photocatalysts was evaluated through hydrogen-evolution reactions under light irradiation from a full-spectrum Xe lamp. Here, 10 vol% ethanol was used as the sacrificial reagent. The Pt cocatalyst, characterized by TEM (Fig. S4), was prepared via the *in-situ* photo-precipitation method using a fixed 0.1 wt% amount. The time courses for H_2 evolution (Fig. 4(a)) and the evolution rates (Fig. 4(b)) for the HT-*T* photocatalysts were determined. Fig. 4(a) reveals that all catalysts show stable rates of hydrogen production. HT-400, with 100% $\text{TiO}_2(\text{B})$, shows the lowest rate of 0.56 mmol/(h·g) (Fig. 4(b)); the activity quickly increases and reaches the highest activity rate of 4.36 mmol/(h·g) on the HT-610 catalyst. After that, the activity decreases, with HT-700, composed of 100% anatase, exhibiting a production rate of 3.31 mmol/(h·g).

The effects of different sacrificial reagents on hydrogen production were investigated over the HT-610 photocatalyst. The amounts of ethanol, methanol, glycerol, and ethylene glycol used in these reaction systems were 20, 5, 10 and 20 vol%, respectively, which were optimized in advance using P25 as a photocatalyst. As shown in Fig. 4(c), both ethanol and methanol show relatively high activities, while glycerol and ethylene glycol show lower activities. The hydrogen-production rate increases in the order: ethylene glycol < glycerol < methanol < ethanol because monohydric alcohols capture excess holes from the surface of TiO_2 more easily, while polyhydric alcohols possess poor abilities for capturing holes. Furthermore, the initial activity in the presence of methanol is the highest among the four, but shows poor stability; the activity began to decrease even after 1 h of reaction time. This is because methanol more easily produces CO as a by-product, which can seriously poison the Pt cocatalyst and decrease its activity [15]. In a word, the highest H_2 -production activity for HT-610 was 4.82 mmol/(h·g), which was obtained in the presence of 20 vol% ethanol as the sacrificial reagent.

To examine the photocatalytic H_2 -evolution stability of the

Table 1
Phase structure of HT-*T* series photocatalysts.

Photocatalyst	Bulk (%) (B, A)	Surface (%) (B, A)
HT-400	(100, 0)	(100, 0)
HT-525	(84, 16)	(55, 45)
HT-575	(56, 44)	(34, 66)
HT-600	(42, 58)	(17, 83)
HT-610	(24, 76)	(0, 100)
HT-650	(17, 83)	(0, 100)
HT-700	(0, 100)	(0, 100)

A: anatase; B: $\text{TiO}_2(\text{B})$.

Fig. 4. Time course for H₂ evolution (a) and photocatalytic H₂-evolution rates (b) for the HT-*T* series of photocatalysts; (c) Photocatalytic H₂-production by HT-610 in the presence of different sacrificial reagents; (d) Recycle testing of the photocatalytic H₂-evolution activity of the HT-610 photocatalyst.

HT-610 photocatalyst, recycling experiments were conducted, as shown in Fig. 4(d). A slight decrease in the amount of produced hydrogen was observed over the entire investigated time, which is probably due to the poisoning effect of the CO by-product adsorbed on the Pt cocatalyst, which cannot totally be eliminated by evacuating the reaction system during the cycling tests.

As described in Table 1, the HT-610 photocatalyst contains 24% of the TiO₂(B) phase in the bulk and 100% of the anatase phase on the surface. Although heterophase junctions were not observed on the surface of the HT-610 photocatalyst, they still function as charge-separation centers in the bulk and increase the final separated charge carriers that diffuse to the surface of the catalyst. On the other hand, in most cases anatase is the best TiO₂ phase for photocatalytic reactions, which is also certified in this research (in Fig. 4(b), HT-700 > HT-400). Therefore, besides efficient charge separation due to the heterophase structure in the bulk, the higher activity of HT-610 also originates from its anatase surface phase. Therefore, the excellent performance of the HT-610 photocatalyst can be attributed to the synergistic effect of the TiO₂(B)/anatase-heterophase junction and the intrinsically high activity of anatase.

3.2.2. Photocatalytic degradation of RhB

The photocatalytic activities of the HT-*T* series of photo-

catalysts were further evaluated through the decomposition of RhB under irradiation from a full-spectrum Xe lamp. Fig. 5(a) shows typical variations in the UV-Vis spectra of aqueous RhB solutions after different illumination times in the presence of HT-600 (58% anatase in the bulk) photocatalyst. As described in the literature [43–45], the decreasing maximum absorption peak at 550 nm during illumination suggests RhB decomposition. The photocatalytic activities for the decomposition of RhB by the HT-*T* photocatalysts are shown in Fig. 5(b). The HT-610 photocatalyst exhibits the best performance and the complete decomposition of RhB was obtained after 60 min. The first-order rate constants for the reaction were calculated according to the Langmuir-Hinshelwood kinetic model (as shown in Fig. 5(c)). The first-order rate constants of the HT-400 to HT-700 photocatalysts are 0.0060, 0.0078, 0.0246, 0.0335, 0.0402, 0.0298, and 0.0219 min⁻¹, respectively. With increasing photocatalyst annealing temperature, the RhB degradation rate first increases and then decreases. These results are in accord with the hydrogen-production activity. It should be pointed out that degradation reactions are more sensitive to the surface area of the catalyst than are H₂-evolution reactions. As a consequence, the rate constants for HT-650 and HT-700 dropped greatly due to their relatively small surface areas. The mixed-phase photocatalyst has a relatively large specific surface area and generates a proper amount of heterophase junc-

Fig. 5. Photocatalytic degradation of RhB with the HT-*T* series of photocatalysts. (a) Typical variations in the absorption spectra of aqueous RhB solutions during illumination in the presence of the HT-600 photocatalyst; (b) Normalized concentration (C/C_0) versus time; (c) Pseudo-first-order kinetic plots. (d) Effect of the EDTA, CH_3OH , and $t\text{-BuOH}$ scavengers, and the oxygen-free system on the photocatalytic degradation of RhB over the HT-610 photocatalyst. (e) Recycling testing for the degradation of RhB over the HT-610 photocatalyst irradiated with light.

tions to promote the separation of photogenerated charges, both of which contribute to the high rate of the photodegradation reaction.

The activated species for the photooxidation reactions mainly include superoxide radicals, hydroxyl radicals, and photogenerated holes [46]. To reveal the activated species in this system, control experiments were performed by adding ethylenediaminetetraacetic acid (EDTA), *tert*-butyl alcohol (*t*-BuOH), and methanol as the trapping reagents for holes, hydroxyl radicals, and oxygen, respectively. An oxygen-free reaction was also performed with continuous nitrogen bubbling during the reaction. Fig. 5(d) shows the photocatalytic degradation of RhB over HT-610 with different trapping reagents; the first-order rate constants for HT-610 alone, under oxygen-free conditions, and in the presence of EDTA, *t*-BuOH, and CH_3OH are 0.0402 min^{-1} , 0.0230 min^{-1} , 0.0029 min^{-1} , 0.0103 min^{-1} , 0.0178 min^{-1} , respectively. The results show that the activity of HT-610 is greatly suppressed in the presence of EDTA, indicating that photogenerated holes are the main activated species in the system. Similarly, the addition of *t*-BuOH also poisons the reaction and sharply decreases the activity, revealing the presence of another important activated species, hydroxyl radicals, in this reaction. The obviously decreased activity of the oxygen-free reaction shows that oxygen-related radicals contribute to the RhB degradation reaction; however, they are not the main active oxidants as the activity was much higher than that in the presence of EDTA. In the presence of methanol, O_2 is quickly consumed and the activity is similar to the oxygen-free reaction. In a word, photogenerated holes are considered to be the main active oxidants in this system, and oxygen-related radicals contribute less so to the reaction.

Furthermore, recycling tests were also performed on HT-610 for the degradation reaction. As shown in Fig. 5(e), the degradation ratio of HT-610 was still maintained above 91.6% after four cycling tests, which shows fairly good catalyst stability. The slight decrease in activity may due to catalyst loss during the sampling process.

3.2.3. Photogenerated charge-carrier behavior of the heterophase junction photocatalyst

Photoelectrochemical measurement is a useful technology to investigate the excitation and transfer of photogenerated charge carriers on photocatalysts. The transient photocurrent responses (*i-t* curves) of HT-400, HT-525, HT-575, HT-600, HT-610, HT-650, and HT-700 were recorded under

Fig. 6. Comparing the transient photocurrent responses of the photocatalysts.

chopped-light irradiation conditions with a 300-W Xe lamp as the light source; the results are shown in Fig. 6. The photocurrent intensity follows the order: mixed-phase TiO_2 > anatase TiO_2 > $\text{TiO}_2(\text{B})$. The results indicate that photogenerated electron-hole pairs are more effectively separated on anatase than $\text{TiO}_2(\text{B})$, and the mixed-phase TiO_2 with the heterophase junction shows even superior behavior. The highest HT-610 current density of HT-610 achieves the most effective charge separation, which is consistent with the results from the photocatalytic reactions.

Besides charge separation, the charge-transfer efficiency is also important for activity. Thus, the series of photocatalysts were further investigated by EIS [47]. The EIS results mainly reflect the charge-carrier transfer kinetic, as presented by charge-transfer resistance (R_{ct}), which is in parallel with a double layer capacitor (C_{dl}) and in series with a solution resistor (R_s) in the equivalent circuit representing the current-flow pathway. Generally, a smaller radius of the Nyquist-plot semi-circle reflects a faster faradaic charge transfer at the interface [48]. As shown in Fig. 7, the smallest arc radius is observed for HT-610 compared to the other photocatalysts, indicating more effective charge-carrier transfer at the interface between the catalyst and the electrolyte. HT-400 and HT-700 show inferior charge-transfer efficiencies that may due to missing heterophase-junction structures.

PL spectroscopy is a highly sensitive tool for studying the photophysics of photogenerated electron-hole pairs, especially charge-recombination information [49,50]. Three typical photocatalysts, HT-400 ($\text{TiO}_2(\text{B})$ phase), HT-610 ($\text{TiO}_2(\text{B})/\text{anatase}$ phases), and HT-700 (anatase phase), were selected for PL spectroscopy measurements. In Fig. 8, the selected three photocatalysts exhibited similar profiles with typical bands centered at 400 nm. Obviously, the PL band of HT-700 shows the highest intensity, while that of HT-400 shows a decreased intensity, and HT-610 exhibits the lowest intensity. Normally, the PL spectrum of TiO_2 originates from the recombination of photogenerated electrons and holes [51–53]. The more recombinations that occur, the higher the intensity of the obtained PL. Therefore, the HT-610 photocatalyst exhibits the best photocatalytic activity because the $\text{TiO}_2(\text{B})/\text{anatase}$ heterophase

Fig. 7. EIS plots over the 10^5 – 0.01 Hz frequency range at an amplitude of 10 mV in the dark.

Fig. 8. PL spectra of the HT-400 ($\text{TiO}_2(\text{B})$), HT-610 ($\text{TiO}_2(\text{B})/\text{anatase}$), and HT-700 (anatase) photocatalysts.

junction effectively inhibits the recombination of photogenerated electron-hole pairs.

In addition to charge-carrier behavior, other influences such as the surface chemical state and light-absorption properties were also investigated. Surface chemical bonding and the elemental binding energies of Ti and O were characterized by XPS, with HT-400, HT-610, and HT-700 chosen as typical samples. As shown in Fig. 9, two $\text{Ti } 2p_{3/2}$ and $2p_{1/2}$ peaks, belonging to the characteristic peaks of Ti^{4+} in TiO_2 [54], are centered at 458.5 and 464.1 eV, 458.4 and 463.9 eV, and 458.4 and 464.1 eV for HT-400, HT-610, and HT-700, respectively. The similar $\text{Ti } 2p$ XPS spectra indicate similar bonding environments of the Ti atoms among the catalysts. The $\text{O } 1s$ spectra can be decon-

Fig. 9. $\text{O } 1s$ and $\text{Ti } 2p$ XPS spectra of the HT-400, HT-610, and HT-700 photocatalysts.

volved into two peaks that represent surface-lattice oxygens (O_L) and surface Ti–OH groups (O_{OH}) using peak-fitting deconvolution [16]. The O_{OH} binding energy (centered around 531.3 ± 0.1 eV) of the different photocatalysts change little, while the O_L position shifts slightly to lower binding energies, from 529.8 to 529.5 eV, indicating that the total bonding environment of the O atoms in HT-610 is a combination of HT-400 and HT-700. This result further proves the formation of heterophase junctions in HT-610. In addition, according to the peak area, the ratio of $O_{OH}/(O_{OH} + O_L)$ for HT-400, HT-610, and HT-700 is 19.9%, 19.0%, and 17.9%, respectively. As the annealing temperature rises, the surface $O_{OH}/(O_{OH} + O_L)$ ratio decreases. The surface O_{OH} may be ascribed to the formation of defects, such as oxygen vacancies (V_o 's) which act as electron donors that improve photocatalytic activity [55]. HT-610 has the lower surface O_{OH} concentration but still shows the highest activity, which is evidence that the heterophase junction is the key issue devoted to the high activity rather than O_{OH} concentration. Furthermore, the UV-Vis diffuse reflection spectra (Fig. S5) show similar absorption edges among the photocatalysts, indicating that light-harvesting ability is also not a crucial issue for high activity.

4. Conclusions

In summary, $TiO_2(B)$ /anatase nanowires with heterophase junction were prepared through a three-step synthesis method. The phase transformation process was monitored by XRD and UV-Raman spectroscopy. It has been found that the phase transformation from $TiO_2(B)$ nanowires to anatase to form the mixed-phase structure of $TiO_2(B)$ /anatase is a surface-preferred phase transformation process. The HT-610 photocatalyst, with an optimized structure containing 24% $TiO_2(B)$ in the bulk and 100% anatase on the surface, exhibits the highest photocatalytic activity, which can produce hydrogen at a rate of 4.82 mmol/(h·g) and degrade RhB with a reaction rate constant of 0.0402 min⁻¹. Charge-carrier behavior during the photocatalytic process reveals that the HT-610 photocatalyst possesses the strongest ability to separate and transport charge, and can also inhibit the recombination of photogenerated electron-hole pairs among the series of photocatalysts. It is speculated that the heterophase junction in the bulk is responsible for the excellent charge kinetic behavior, while the surface phase of anatase takes control of the high surface reaction activity.

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Graphical Abstract

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Fabrication of TiO₂(B)/anatase heterophase junctions in nanowires via a surface-preferred phase transformation process for enhanced photocatalytic activity

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TiO₂(B)/anatase-heterophase-junction nanowires were prepared via a surface-preferred phase-transformation process. The high charge-separation efficiency, due to the heterophase junction (bulk), and the excellent surface reaction of anatase (surface) both contribute to the enhanced photocatalytic activity.

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基于表面优先型相变过程构建 TiO₂(B)/锐钛矿异相结纳米纤维及其光催化性能

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摘要: 近年来, 随着光催化研究的逐渐深入, 人们发现有效的电荷分离往往是提高半导体光催化材料活性的关键步骤. 其中, 在多晶半导体材料中构建异相结结构是提高其活性的最有效途径之一. 在 TiO₂ 四种稳定晶相中, TiO₂(B) 是发现最晚且研究较少的, 并且目前对它的研究主要集中于锂离子电池方面, 而在光催化领域中研究较少. 然而, 该材料特殊的晶相结构带来的各向异性和较大的比表面积都表明其在光催化领域同样具有潜在应用前景. 本文通过三步合成法, 包括水热过程、离子交换、退火过程, 研究了 TiO₂(B) 向锐钛矿转变的相变过程, 同时构建了具有 TiO₂(B)/锐钛矿异相结结构的纳米线. 在材料退火过程中, 通过 X 射线衍射 (XRD) 和表面灵敏的紫外拉曼光谱 (UV-Raman) 分别监测了样品体相和表面

相的晶相结构变化过程. 发现了 XRD 与 UV-Raman 结果的不同步性. 同时, 通过对样品扫描电镜和透射电镜的形貌表征可知, $\text{TiO}_2(\text{B})$ 纳米线在退火过程中部分结构发生坍塌, 且逐渐转化成锐钛矿相, 而相变过程很可能首先发生在坍塌结构处. 高分辨电镜结果表明, 纳米线中同时存在着 $\text{TiO}_2(\text{B})$ 结构和锐钛矿结构, 并形成很好的异相结. 根据相结构变化和形貌观察结果, 我们推测 $\text{TiO}_2(\text{B})$ 向锐钛矿转变过程为表面优先型的相变过程, 即材料表面相先于体相先发生相变过程, 由此得到的材料表面相含有较多的锐钛矿, 而体相中含有较多的 $\text{TiO}_2(\text{B})$. 为了验证构建的异相结材料的光催化活性, 采用光催化产氢和降解污染物双重反应对样品进行了表征. 结果发现, 单独的 $\text{TiO}_2(\text{B})$ 材料活性较锐钛矿材料相差较多, 但其混相结构能较大幅度地提高光催化活性. 其中, 通过逐渐退火过程形成的体相为 24% 的 $\text{TiO}_2(\text{B})$ 、表面为 100% 的锐钛矿相的催化剂活性最高, 其产氢活性可达 $4.82 \text{ mmol}/(\text{h} \cdot \text{g})$, 降解污染物反应速率常数为 0.0402 min^{-1} . 采用光电流测试、电化学阻抗和荧光光谱表征了材料在光催化过程中的载流子动力学行为. 结果表明, 最优活性的催化剂中体相的异相结结构对光生电荷的分离、传输都起到良好地促进作用, 而表面的锐钛矿相主要负责高活性的表面反应, 二者共同对高活性的光催化反应起到推动作用.

关键词: $\text{TiO}_2(\text{B})$ /锐钛矿; 异相结; 表面优先; 光催化; 相变

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根据美国科睿唯安 (Clarivate Analytics) 近日公布的2017年度《期刊引证报告》(Journal Citation Reports, JCR), *Chinese Journal of Catalysis* (《催化学报》) 的最新SCI影响因子 (IF) 为 3.525 (2016年为 2.813), 在国内化学类期刊中名列前茅; 在国际化学工程学科137种期刊中排名第26位, 排名居前18.98%, 位居Q1区. 近年, 《催化学报》的学术质量及影响力不断提升, 这是中国化学会催化委员会委员、编委、审稿专家、作者和读者长期不懈努力和共同支持的结果. 衷心感谢各位老师对期刊宣传、投稿原创性高质量稿件、学术质量把关以及组约优秀稿件、组办高水平专刊等方面所给予的持续和巨大的支持!

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