Plasmon-assisted partially reduced TiO$_2$ nanotube arrays for photoelectrochemical water splitting

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Abstract

The partially reduced TiO$_2$ nanotube arrays (R-TNTs) has been extensively investigated for photoelectrochemical (PEC) water splitting. However, the severe charge recombination and limited light harvesting hinder their photocurrent output. Herein, we present a strategy for prepared R-TNTs photoanode which can facilitate the charge separation and transfer simultaneously via formation of homojunction induced by the Ti$^{3+}$ self-doping and oxygen vacancies. Additionally, we deposited plasmonic Au nanoparticles on the R-TNTs surface to extend the light absorption in visible region and accelerate the charge transfer at the solid-liquid interface via plasmonic effect. As a result, in PEC water splitting experiments, the optimized Au modified R-TNTs (Au@R-TNTs) photoanode exhibits a photocurrent density of 1.64 mA·cm$^{-2}$ with solar-to-hydrogen conversion efficiency up to 0.55% under AM 1.5 G illumination, 4-fold and 2-fold higher than that of the pristine TNTs, respectively. The photocatalytic activity in the visible region was dramatically improved. The exploration of interfacial carrier dynamics in homojunction and the investigation of the synergistic effects of plasmonic enhanced photocatalyst provides guideline for designing solar energy harvesting devices.

1. Introduction

Developing clean and renewable energy has attracted great interest due to the excessive consumption of fossil fuels [1]. Converting solar energy into chemical energy as a form of H$_2$, is an attractive approach to solve the energy challenge [2]. Photoelectrochemical (PEC) water splitting is a promising and environmentally friendly method to realize solar energy utilization [3]. Several kinds of semiconductor catalysts can be applied to PEC water splitting, such as TiO$_2$ [4–6], Fe$_2$O$_3$ [7, 8] and BiVO$_4$ [9, 10]. Among all these candidates, TiO$_2$ is more promising than others due to its compatible band-edge positions, resistance to photo corrosion, high photocatalytic activity, low cost and low toxicity [4, 11–14]. However, the large band gap of TiO$_2$ [15](i.e., 3.2 eV for anatase and 3.0 eV for rutile) limits its absorption only in ultraviolet (UV) region, and its poor absorption in the visible region leads to low photoelectric conversion efficiency. Moreover, the severe charge recombination in the bulk results in an inefficient utilization of photogenerated carriers.

Several strategies, such as electrochemical reduction [16–18], doping of metals or nonmetals (Ni, C, N) [19–21], coupling with another semiconductors (ZnO, CdS) [22, 23] and photosensitization of dyes [24, 25] have been used to accelerate charge transfer and enhance the light absorption. However, there is still challenge to simultaneously enhance charge transfer and the light harvesting. In this work, two methods were combined to realize accelerating the charge transfer and enhancing the light absorption at the same time. Firstly, Ti$^{3+}$ and oxygen defects were introduced into the interface of TiO$_2$/reduced TiO$_2$ nanotube arrays (R-TNTs), forming a homojunction for enhancing the carrier separation and accelerating the charge transfer. And at the same time, the R-TNTs shows slight enhancement in the visible region due to the increased carrier density. Secondly, Au
nanoparticles (NPs) were deposited on the TiO₂ surface to significantly enhance the light absorption in visible range through surface plasmon resonance (SPR) effect and accelerate the charge transfer at the solid-liquid interface. The optimized Au@R-TNTs photoanode achieves a photocurrent density of 1.64 mA · cm⁻² with solar-to-hydrogen conversion efficiency up to 0.55%, which is 4-fold and 2-fold increase compared to that of the pristine TNTs. The Mott-Schottky analysis confirms that the carrier density increase from 1.43 × 10²⁰ cm⁻³ in TNTs to 4.00 × 10²⁶ cm⁻³ in Au@R-TNTs electrode. The synergistic effects of Au@R-TNTs can effectively enhance both the photogenerated electron-hole separation and transfer via formation of homojunction and the light absorption in visible region via SPR effect, it is a promising way for improving photocatalytic activity.

2. Experimental section

2.1. Materials
Ammonium fluoride (NH₄F), ethylene glycol (EG), sodium sulfate (Na₂SO₄) and chloroauric acid (HAuCl₄·4H₂O) were purchased from Aladdin. Polyvinylpyrrolidone (PVP), ethanol and potassium hydroxide (KOH) were purchased from Kermel. All reagents were analytical grade and used without further purification. Deionized water (DIW) with a resistivity of 18.2 MΩ · cm was purified through a Milli-Q water system.

2.2. Fabrication of TNTs
The preparation process of the photoanodes is illustrated in Scheme 1. The vertically oriented TiO₂ nanotube arrays (TNTs) were produced through a two-step anodizing process in a two-electrode electrochemical cell at room temperature, using a Ti foil (1 × 2 cm², 0.2 mm, 99.9% purity, Baoji) as the working electrode and a platinum foil as the counter electrode. The Ti foil was first anodized in the electrolyte of ethylene glycol with 2 vol% DIW and 0.5 wt% NH₄F under 60 V for 0.5 h. Then, it was anodized for another 1 h to get TNTs after removed the formed layer, and then annealed at 450 °C for 2 h with a heating rate of 5 °C/min. The as-prepared photoanode is named as pristine TNTs.

2.3. Electrochemically doping
Electrochemical reductive doping was conducted in a three-electrode system at room temperature with the prepared TNTs as working electrode, Pt foil as counter electrode and Ag/AgCl as reference electrode. Ti⁴⁺ is reduced to Ti³⁺ by electron transfer from the cathode during this process [17]. TiO₂ on top of Ti foil was first reduced. We find that the TNTs prepared in 1 M Na₂SO₄ aqueous solution through potentiostatic cathodic reduction at −1.0 V versus Ag/AgCl for 30 min (denoted as R-TNTs) shows the best catalytic properties.

2.4. Deposition of Au NPs
The deposition of Au NPs on the R-TNTs was achieved through photo-reduction method. The Au³⁺ precursor solution was prepared with 2 ml HAuCl₄ aqueous solution (1 mM), 0.2 g PVP (K₃,0, MW = 58 000) and 18 ml ethanol. The as-prepared R-TNTs were immersed into this Au³⁺ precursor solution and irradiated under a 300 W xenon lamp (PLS-SXE300) for 2 to 12 min The optimal time for photo-reduction is 8 min and we denoted it as Au@R-TNTs. (The exploration for optimal condition of each step is shown in figure S2 is available online at stacks.iop.org/MRX/6/1250h9/mmedia).
2.5. Characterization

The morphology and structure of the photoanode materials were characterized by field-emission scanning electron microscopy (FESEM) (Hitachi S-4800, 5 kV). Elemental analysis was carried out by energy dispersive spectroscopy (EDS) at 15 kV. Transmission electron microscopy (TEM) image and high-resolution TEM image were collected at an acceleration voltage of 200 kV to further confirm the size and distribution of the Au NPs. The x-ray diffraction (XRD) measurements were carried out on a Bruker D8 Advanced diffractometer. Optical absorption measurements were obtained by using a UV-2700 UV–vis spectrophotometer. X-ray photoelectron spectroscopy (XPS) and ultraviolet photoelectron spectroscopy (UPS) analysis of the materials were carried out on a Thermo ESCALAB 250XI system.

2.6. PEC water splitting measurements

PEC water splitting measurements were performed in a three-electrode configuration with the samples as the working electrode, a platinum foil (1 × 1 cm²) as the counter electrode and a saturated Ag/AgCl as the reference electrode in a 1.0 M KOH (pH = 13.9) aqueous solution. 500 W xenon lamp (CEL-S500) equipped with AM 1.5 G simulated solar irradiation was used as the light source during the whole experiment. The power intensity of the light and the illuminated area of the photoanode materials were fixed to 100 mW · cm⁻² and 1 cm², respectively. The method for transformation the potential versus Ag/AgCl electrode to hydrogen electrode (RHE) was shown in SI.

The liner sweep voltammetry curves (LSVs) were tested under the condition of both light and dark illumination. Electrochemical impedance spectroscopy (EIS) was performed to explore the conductivity of the photoanodes at open circuit voltage over a frequency range from 10⁻¹ to 10⁵ Hz. The I-t curve was evaluated under chopped light irradiation for several light/off cycles at 1.23 V versus RHE and the stability test was evaluated at 1.23 V versus RHE for 2 h. The Mott–Schottky plots were obtained at frequency of 1 kHz.

3. Results and discussion

To reduce the severe charge recombination in the bulk and improve the limited light harvesting in the TiO₂ photoanode, we propose a structure that combining R-TNTs/TNTs homojunction with Au particles to simultaneously solve the above issues. In order to verify this hypothesis, we first study crystal structure and surface morphology of R-TNTs and Au@R-TNTs. Figure 1 (a) shows the XRD patterns of the intermediate products (pristine TNTs and R-TNTs) and final product (Au@R-TNTs). The diffraction peaks of all photoanodes are consistent with the anatase TiO₂ (No. 21-1272). However, the patterns of Au particles are not detected due to the low content [26]. To further study this nanocomposite anode, the corresponding SEM and TEM images were measured. The surface morphology of Au@R-TNTs as shown in figures 1(b) and S1c reveal
that Au NPs were uniformly deposited on the porous surface of the R-TNTs. The successful deposition of Au NPs was further confirmed by TEM images. Figure 1(c) shows that Au NPs deposited on the surface and inner wall of nanotube arrays. The high-resolution TEM of square region in figure 1(d) reveals that the lattice distance of 0.23 nm matches with the interspacing of Au \{111\} facets \[15, 26, 27\]. All the data shows that Au@R-TNTs electrode was successfully prepared through our strategy.

XPS and UPS measurements were performed to analyze the effect of electrochemical reduction and Au NPs deposition on the modification of electronic structure and energy band structure. Figure 2(a) shows the fine XPS spectrum of Ti 2p. The two broad peaks centered at approximately 458.7 eV and 464.5 eV can be split into four peaks of Ti 2p_{3/2} (Ti^{4+}), Ti 2p_{1/2} (Ti^{4+}), Ti 2p_{3/2} (Ti^{3+}) and Ti 2p_{1/2} (Ti^{3+}) upon fitting \[16, 18, 28\]. According to the specific information listed in table S1, the R-TNTs (1.54) shows the lower ratio of Ti^{4+}/Ti^{3+} than pristine TNTs (2.16), which indicates that part of Ti^{4+} has been reduced into Ti^{3+} during the procedure of electrochemical reduction \[29\]. For the O1s XPS spectrum (figures 2(c) and (d), table S1), the content of oxygen vacancies (Ti-OH, 531.5 eV) in R-TNTs has increased after electrochemical reduction, which is consistent with that of Ti 2p spectra. Therefore, we can conclude that the high concentration of Ti^{3+} and oxygen vacancies have been successfully introduced into the TiO2 during the process of electrochemical reduction.

The UPS spectra suggest that the electrochemical reduction does not affect the Fermi edge position while the low cutoff edge does shift to a lower energy position compared with the pristine TiO2 photoanode. The position and specific value of Fermi edge and cutoff edge were shown in figure 3(b) \[30, 31\]. According to the following equation:

\[
\varphi = h\nu - (E_{\text{Fermi}} - E_{\text{Cutoff}}); \quad E_F = E_{\text{Vacuum}} - \varphi
\]

where \(\varphi\) is the work function, \(h\nu\) is the energy value of an UV light source (\(h\nu = 21.2\) eV), \(E_{\text{Cutoff}}\) and \(E_{\text{Fermi}}\) are the value of cutoff edge and Fermi edge. \(E_{\text{Fermi}}\) corresponds to the abscissa value of the midpoint of the Fermi edge curve. \(E_{\text{Cutoff}}\) is determined by the intersection of the tangent of the cutoff edge and the baseline \[32\]. The \(E_{\text{Fermi}}\) of pristine TNTs and R-TNTs samples are both 26.5 eV, while the \(E_{\text{Cutoff}}\) of pristine TNTs and R-TNTs samples are 10.2 eV and 9.6 eV, respectively. And then the work function of pristine TNTs (4.9 eV versus vaccum) and R-TNTs (4.3 eV versus vaccum) samples are obtained according to the equation (1). These findings indicate that the work function of R-TNTs (4.3 eV versus vacuum) is lower than that of pristine TiO2 (4.9 eV). Combined with the valence band maximum and bandgap obtained from the valence band spectra (figure 3(a)) and UV–vis spectra (figure 4(b)), respectively, we can obtain the schematic energy band diagram of Au@R-TNTs.

Figure 2. (a) and (c) XPS spectra of pristine TNTs; (b) and (d) XPS spectra of R-TNTs.
Figure 3. (a) Valence band spectra of pristine TNTs and R-TNTs obtained from XPS data; (b) UPS spectra of pristine TTNts and R-TNTs; (c) Schematic illustration of charge transfer process of the Au@R-TNTs.

Figure 4 (a) Mott-Schottky curves and (b) UV–vis spectra of three photoanodes.

as shown in figure 3(c). According to the diagram, the R-TNTs migrates downward and the pristine TNTs migrates upward to get balanced for the Fermi energy when they are in contact [33]. As a result, the R-TNTs combined with pristine TNTs forming an n+–n homojunction that can facilitate the photogenerated electron-hole separation and transfer. When Au NPs deposited on the homojunction, the hot electron can inject into the conductive band of R-TNTs to reduce H+ to form H2. At the same time, the charge transfer could be promoted due to the built-in electric field. As a result, the formation of n+–n homojunction and the deposition of Au NPs was proposed to provide an effective way for photogenerated electron-hole separation and transfer, which can avoid the carrier recombination and improve PEC catalyst activity.

The Mott–Schottky curves (figure 4(a)) and UV–visible absorption spectra (figure 4(b)) were measured to analyze the intrinsic electronic properties and light absorption of the photoanodes. The Mott–Schottky curves in figure 4(a) shows that all the three photovoltaic electrodes have a positive slope, which is in accordance with the n-type semiconductor. The inset shows an enlargement of the region circled with a green dotted line. The carrier
density ($N_d$) and flat band potential ($V_{FB}$) are calculated following the equation [34–36]:

$$N_d = \frac{2}{\varepsilon_0 \varepsilon_r \varepsilon_0} \left[ \frac{d}{dV} \left( \frac{C}{V} \right) \right]^{-1}$$

(2)

$$\frac{1}{C_{SC}^2} = \frac{2}{\varepsilon_0 \varepsilon_r A \varepsilon_0 N_d} (V - V_{FB} - kT / \varepsilon_0)$$

(3)

where $N_d$ is the carrier density, $e_0$ is the electron charge, $\varepsilon_r$ is the relative permittivity of anatase TiO$_2$ ($\varepsilon_r = 48$), $\varepsilon_0$ is the permittivity of vacuum, $V$ is the applied bias versus RHE, $C_{SC}$ is the space charge capacitance in the semiconductor and $A$ is reaction area. When the capacitance of the Helmholtz layer can be neglected, $C_{SC}$ is equal to $C$. The value of $C$ is obtained according to the following equation:

$$C = -1/\omega Z_i$$

where $\omega = 2\pi f$ is the angular frequency, $Z_i$ is the imaginary component of the impedance. Through the electrochemical test of Mott–Schottky, the data of $Z_i$ and $f$ are recorded as a function of potential. And then the corresponding value of $C_{SC}$ is obtained with the change of voltage. The specific information of flat band potential ($V_{FB}$) and carrier density ($N_d$) for the three photoanodes are collected in table S2. Obviously, the carrier density increases dramatically from $1.43 \times 10^{20}$ cm$^{-3}$ in pristine TNTs to $1.04 \times 10^{25}$ cm$^{-3}$ after the process of electrochemical reduction, which mainly due to the efficient charge transfer and separation based on the formed homojunction. Furthermore, the carrier density increases by an order of magnitude ($4.00 \times 10^{26}$ cm$^{-3}$) after the deposition of Au NPs owing to its SPR effect and the enhanced charge transfer ability at the solid–liquid interface.

UV–visible absorption spectra in figure 4(b) shows that R-TNTs has higher absorption than that in pristine TNTs, which mainly owes to their lower bandgap [37] and free-carrier absorption [38]. After the uniform deposition of Au NPs, the Au@R-TNTs photoanode shows strong absorption in the visible light region at around 525 nm. This result demonstrates that the presence of Au NPs expands the absorption range of pristine TNTs to visible light due to the plasmonic effect.

The PEC performances were measured to confirm the rationality of the formed homojunction and the function of Au NPs. Figure 5(a) shows that the dark current density of Au@R-TNTs photodevice from 0.2 V to 1.4 V (versus RHE) is nearly zero. The photocurrent density of pristine TNTs is 0.41 mA·cm$^{-2}$ at a potential of 1.23 V versus RHE, which is comparable to the photocurrent density of TiO$_2$ nanotube arrays reported in previous articles [39]. The photocurrent density of R-TNTs photoanode at a potential of 1.23 V versus RHE is 1.13 mA·cm$^{-2}$, more than twice as high as that of pristine TNTs. After the decoration of Au NPs, the photocurrent density of Au@R-TNTs photoanode achieves 1.64 mA·cm$^{-2}$ at 1.23 V versus RHE, which is nearly four times that of pristine TNTs. The photoconversion efficiency (figure 5(b)) is calculated by the following equation:

$$\eta(\%) = \frac{(1.23 - V_{app})}{P_{light}} \times 100$$

(4)

where $V_{app}$ is the applied external potential versus RHE, $I$ is the photocurrent density measured in this experiment. $P_{light}$ is the power density of the xenon lamp used in the PEC water splitting test. The maximum photoconversion efficiencies for the photoanodes of pristine TNTs, R-TNTs and Au@R-TNTs are 0.23% (at 0.52 V versus RHE), 0.40% (at 0.67 V versus RHE) and 0.55% (at 0.70 V versus RHE), respectively. The enhanced photocurrent density and photoconversion efficiency demonstrate that the formed homojunction and Au NPs synergistically accelerate the charge transfer and increase the availability of visible light, leading to an improved photocatalytic performance [15, 40].

EIS was measured to further explore the interfacial properties between the photoelectrodes and the electrolyte. Figure 5(c) shows the Nyquist plots for the pristine TNTs, R-TNTs and Au@R-TNTs under AM 1.5 G illumination. And the equivalent circuit model is in the inset of figure 5(c) [16]. Obviously, the diameter of the semicircle for R-TNTs is much smaller than that of pristine TNTs, indicating that the formed homojunction has greatly enhanced the charge transfer by the introduced built-in electric field in the homojunction. And the diameter of the semicircle for Au@R-TNTs is the smallest of them all, which further confirms that Au NPs accelerate the charge transfer at the solid–liquid interface.

IPCE measurement was carried out to investigate the photo–conversion efficiency and the calculated equation was shown in SI. Compared with pristine TNTs, the R-TNTs shows slight enhancement in the UV region and photo-activity in visible region. Moreover, after Au NPs deposition, the IPCE of the Au@R-TNTs in the wavelength range from 480–550 nm shows maximum at 520 nm, much higher than that of the pristine TNTs. The increased IPCE of Au@R-TNTs shows agreement with their extended absorption in visible region (figure 4(b)), which confirms that the plasmonic enhancement can contribute to the PEC efficiency [15]. The Au@R-TNTs photoanode was tested at 1.23 V versus RHE under on-off cycle illumination conditions to examine the capability of photoresponse. As shown in figure 5(e), the current density for Au@R-TNTs goes through a sharp increase from several $\mu$A·cm$^{-2}$ in the dark to mA·cm$^{-2}$ scale in the presence of light, which
means that the Au@R-TNTs photoelectrode possesses excellent sensitivity to light illumination. Finally, we measured the performance of optimized photoanode at 1.23 V versus RHE under AM 1.5 G illumination to examine the stability. Figure 5 shows that the photocurrent density of Au@R-TNTs is almost unchanged during 2 h, indicating that the Au@R-TNTs in the electrolyte have good PEC stability under long time irradiation.

4. Conclusions

In summary, we have developed an approach to form homojunction in R-TNTs through introducing the Ti$^{3+}$ and oxygen vacancy. The built-in electric field in the homojunction accelerates the charge separation and transfer. Moreover, by incorporation with Au-NPs, the light absorption of Au@R-TNTs were extended from the UV region into the visible region. The SPR effect of Au NPs combined with the fast charge transfer ability of R-TNTs, leads to the enhanced solar-to-hydrogen conversion efficiency. The photocurrent density of Au@R-TNTs photoanode achieves about 1.64 mA · cm$^{-2}$ at 1.23 V versus RHE under AM 1.5 G illumination, which is
nearly four times that of pristine TNTs. The present work provides a simple and low-cost strategy to effectively improve the PEC catalytic performance for potential solar hydrogen production.

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Conflicts of interest

There are no conflicts to declare.

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