

Light energy accumulation using Ti/RuO₂ electrode as capacitor

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Abstract In the present study, the possibility to use Ti/RuO₂ electrode as capacitor for storage of photoelectrons generated under UV irradiation in Ti/TiO₂ photoelectrode has been investigated. A light-sensitive TiO₂ layer has been formed by means of anodizing Ti electrode in the solution of 0.5 M H₂SO₄. A layer of RuO₂, exhibiting the properties of electrochemical capacitor, has been formed by means of thermal decomposition of RuOHCl₃ also on Ti substrate. The photocharging capability of RuO₂ has been studied by means of short-circuiting Ti/RuO₂ electrode with Ti/TiO₂ photoelectrode in deaerated solution of 0.1 M KOH. It has been shown that the intensity of photocurrent flowing from Ti/TiO₂ to Ti/RuO₂ electrode depends mainly on the potential of the latter. Maximum value of photocurrent density was ~180 μA cm⁻², which corresponded to maximum value of photon-to-electron conversion efficiency (IPCE) of about 60%. The amount of photogenerated charge Q_{ph} , which can be stored, depends on the capacitance of RuO₂ coating. Under the conditions of the experiment, Q_{ph} ranged from ~35 to ~50 mC, which corresponded to a specific charge of RuO₂ coating ranging between ~20 and ~30 mAh g⁻¹.

Keywords UV light · Energy conversion · Energy storage · Capacitance · TiO₂ · RuO₂ electrode

Introduction

Solar energy represents one of the most attractive and viable renewable energy resources [1]. Though ~15% efficiency of light-to-electric energy conversion has been already achieved

using single-crystal silicon-based photovoltaic devices, their production and installation is expensive and complicated. So harvesting of photons with greater efficiency and at lower cost still remains the main challenge in the sphere of solar energy utilization [1].

Separation of photogenerated charge carriers represents an important issue in electrochemistry of semiconductors as it has crucial influence on the efficiency of light energy conversion devices. Effective conversion of photogenerated charge carriers' energy into chemical energy facilitates the charge separation, enhances the photoconversion efficiency, and is also interesting from the viewpoint of light energy storage [2, 3], photo rechargeable batteries [4, 5], and protection of metals against corrosion [3, 6, 7]. Various semiconductor modification methods have been proposed for maximizing the efficiency of charge separation and energy conversion [1]. TiO₂-based systems are known to be the most stable and photo corrosion-resistant in aqueous solutions and therefore are being widely investigated due to their potential application in the photocatalytic splitting of water [8–11]. According to a recent review [1], modification of TiO₂ with Pt, Pd, Rh, Ru, Au, Ag, Sn, and Ni has been investigated and in many cases some increase in the efficiency of photocatalytic reactions has been confirmed. Oxides represent another type of materials, which can be coupled with semiconductors to accumulate the photo-generated charge. The main principle here is to use multivalent metal oxides with sufficient conductivity to ensure the facile electron transfer and feasibility of redox transition to utilize the photogenerated electrons or holes. Essential requirement for the proper functioning of light energy conversion and storage system is suitable redox potential of electrochemical reactions which are involved in the process of accumulation of photogenerated charge carriers [2]. Accumulation of photoelectrons within the oxide, or the so-called photocharging, is believed to

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proceed via the partial reduction of metal cation that is accompanied by the intercalation of protons or other cations from the electrolyte to compensate the negative charge, which can generally be described by the equation as follows [2, 3, 6, 12, 13]:



Modification of TiO₂ with SnO₂ [6], Fe₂O₃ [12], WO₃ [7], and MoO₃ [13] has been studied. In all the above cases, the possibility of photogenerated electron storage was confirmed; however, the efficiency of the process was far from desirable. More promising results were obtained by Wang et al. [2], who investigated the photo-chargeability of titanium/vanadium oxide composites. The authors have reported maximum IPCE of 37.1%; however, the specific photocharging capacity of composite TiO₂/V₂O₅ electrodes was rather low, about 2.8 C g⁻¹ (~0.8 mAh g⁻¹). Very similar specific charge values were reported in [7] for TiO₂/WO₃ composite films.

In the present study, the photocharging capability of positively charged Ti/RuO₂ electrode short-circuited with Ti/TiO₂ photoelectrode was explored. In other words, an attempt has been made to increase the photogenerated charge separation efficiency, using Ti/RuO₂ electrode as acceptor for photoelectrons. Ruthenium dioxide has the highest metallic-type electric conductivity among the oxides [14] and is a well-known charge-storage material with high specific capacitance. Values up to 1,500 F g⁻¹ have been reported for RuO₂ deposited on the activated carbon [15]. To the best of our knowledge, the capability of Ti/RuO₂ to accumulate the photogenerated charge from Ti/TiO₂ photoelectrode has not been studied so far.

Experimental

Preparation of the electrodes

Fabrication of photosensitive titania films on the surface of Ti electrode by means of high voltage anodization is a well-known process, which has been intensively studied [16, 17]. In our study, photosensitive layer of TiO₂ on titanium substrate was formed by means of anodizing Ti plate (1 × 1 cm) in the solution of 0.5 M H₂SO₄ for 5 min at anodic current density $i_a \approx 50 \text{ mA cm}^{-2}$ and the anode–cathode voltage ranging between 150 and 180 V. The anodization procedure was followed by heat treatment for 90 min at 400 °C in air. Anodized Ti plate had gray appearance. SEM image of the anodically formed titania is presented in Fig. 1. Image reveals the surface with disordered distribution of pores with the size in submicrometer scale. Formation of pores resulted from high cell voltage applied

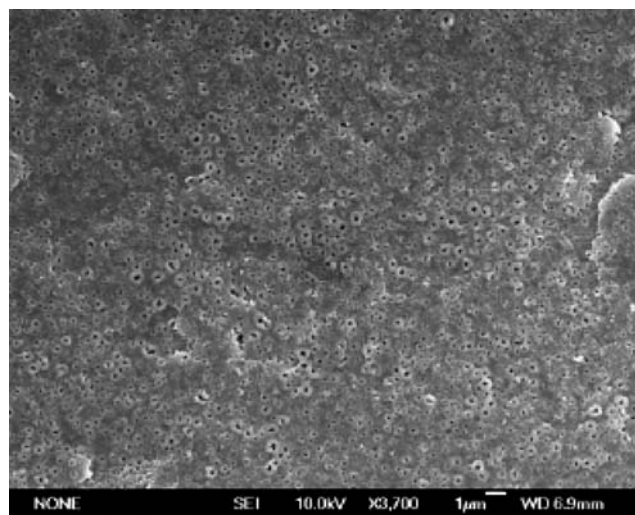


Fig. 1 SEM image of titanium anodized in 0.5 M H₂SO₄ for 5 min at $i_a \approx 50 \text{ mA cm}^{-2}$ and anodizing voltage of 150–180 V

for anodizing, which was higher than sparking discharge voltage of TiO₂, i.e., >100 V [17, 18]. Visual inspection of the samples was performed using a scanning electron microscope (JSM-6700FT, JEOL).

RuO₂ layer on titanium substrate was formed by means of thermal decomposition of RuOHCl₃, as described in Ref. [14], according to the procedure as follows. A layer of RuOHCl₃ solution in the mixture of 1 M HCl and isopropanol (1:1) was painted onto titanium surface (1 cm²) and heated at 400 °C for 10 to 15 min to ensure the adhesion. The process was repeated five times and then the electrode was heated additionally for 1 h at 400 °C. The amount of RuO₂ deposited on the electrode was about 0.45 mg cm⁻² which corresponds to oxide layer thickness of ~0.6 µm.

Photoelectrochemical measurements

Photoelectrochemical measurements were performed in a three-electrode electrochemical cell supplied with quartz window. Schematics of electrochemical cell and electric equivalent circuit are shown in Fig. 2. When the photo-induced electron transfer from Ti/TiO₂ to Ti/RuO₂ was investigated, these electrodes were connected through the 2 Ω resistance for photocurrent I_{ph} measurement. The potential drop across this resistance did not exceed ~0.2 mV at maximum values of I_{ph} , so the influence of 2 Ω resistance on I_{ph} can be neglected. Analog–digital converter (ADC) connected to PC was used for I_{ph} measurement. The values of open-circuit potential $E_{i=0}$ of Ti/TiO₂ and Ti/RuO₂ electrodes and also short-circuited electrodes were measured with respect to hydrogen electrode in a working solution (RHE) using potentiostat–galvanostat ПИИ-50-1 (Russia) connected to a PC through ADC interface.

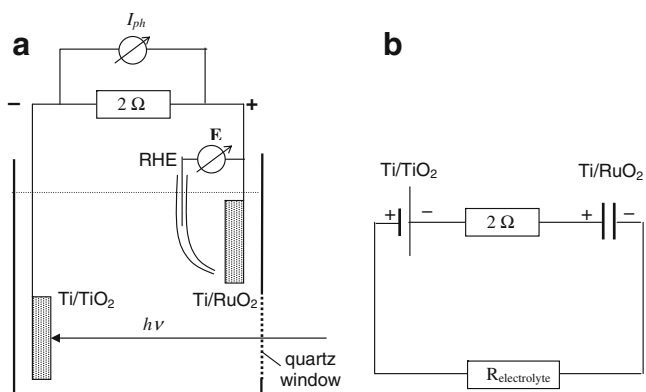


Fig. 2 Schematics of electrochemical cell (a) and electric equivalent circuit (b), where Ti/TiO₂ electrode acts as photo power source and Ti/RuO₂ electrode as capacitor

Potential values in the text refer to RHE scale. The relationship between RHE and SHE is given by:

$$E_{\text{RHE}} = E_{\text{SHE}} - 0.059\text{pH} \quad (2)$$

TiO₂ surface was illuminated by 120 W Hg lamp. The light was passed through monochromator MДP-12 (JIOMO, Russia) set at 365 nm. The light intensity at the titania electrode surface was about 1 mW cm⁻² as measured with LM2 power meter (Carl Zeiss, Jena, Germany). It is important to note that intensity of 1 mW cm⁻² is similar to that of sunlight UV [13]. The area of TiO₂ exposed to the solution was ~0.5 cm².

Platinum wire was used as counter electrode in voltammetric and galvanostatic charging measurements.

Solutions of 0.5 M H₂SO₄ (pH 0.65) and 0.1 M KOH (pH 12.8) were used for investigations. Solutions were prepared from reagents of analytical grade and triply distilled water. To remove dissolved oxygen the solutions were bubbled with pure argon for 30 min prior to measurements. Argon was also used to maintain the inert atmosphere over the solutions during the measurements. All measurements were carried out at room temperature.

Incident photon-to-electron conversion efficiency (IPCE) was calculated according to following equation [1]:

$$\text{IPCE}(\%) = \frac{1240 \times I_{\text{ph}}}{\lambda \times I_{\text{int}}} \times 100\% \quad (3)$$

where I_{ph} is the short-circuit photocurrent density (mA cm⁻²), I_{int} is the incident light intensity (mW cm⁻²) and λ is the wavelength (nm).

Results and discussion

Investigations of Ti/RuO₂ electrode charging in 0.5 M H₂SO₄

Voltammetric behavior of Ti/RuO₂ electrode in the solutions of 0.5 M H₂SO₄ and 0.1 M KOH is shown in

Fig. 3a. Such capacitor-type response, where current density is almost independent of potential over a wide range of E values, is typical for RuO₂ electrodes [15, 19–21]. The mechanism of charge storage at RuO₂/0.5 M H₂SO₄ interface has been discussed recently in [22]. One can see from Fig. 3a that when anodic or cathodic current is being passed through the Ti/RuO₂ electrode within 0.2 V–1.4 V its potential turns more positive or negative, respectively. Thus, from the character of Ti/RuO₂ potential change, one can judge about the direction of current flowing through the electrode. Presuming that at 1.4 V the Ti/RuO₂ capacitor is fully positively charged, the connection of electron source to such electrode should lead to discharging or negative charging of the capacitor and at 0.2 V the capacitor could be treated as being fully negatively charged. At E < 0.2 V, reduction of RuO₂ to Ru(OH)₃ or even to Ru becomes possible [23]. Reduction of oxygen dissolved in the solution is possible at E < 0.8 V [23, 24]. At E > 1.4 V, oxygen evolution begins.

First, the possibilities of electron transfer between Ti/TiO₂ photoelectrode and Ti/RuO₂ were investigated in acidic media, i.e., in the solution of 0.5 M H₂SO₄. The open-circuit potential E_{i=0} of anodized titanium in 0.5 M H₂SO₄ usually was in the range between 0.45 and 0.6 V, whereas that of Ti/RuO₂ electrode was within 0.75–0.85 V, which corresponds to halfly charged or discharged state of the capacitor. As one can see from the inset in Fig. 3, Ti/TiO₂ is passive at E > 0.6 V and should attain the value of Ti/RuO₂ electrode potential upon short-circuiting. Yet, when these electrodes were short-circuited, the potential of RuO₂ electrode usually shifted negatively. The shift of

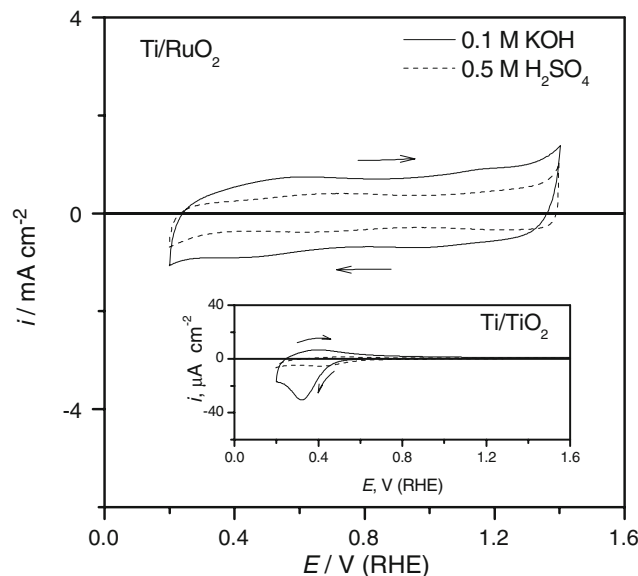


Fig. 3 Cyclic voltammograms of Ti/RuO₂ and Ti/TiO₂ (inset) electrodes in the solutions of 0.5 M H₂SO₄ and 0.1 M KOH, ν = 20 mV s⁻¹; 20 °C

$E_{i=0}$ of RuO₂ towards more negative values points to some flux of electrons from Ti/TiO₂ to Ti/RuO₂ upon short-circuiting. Moreover, it was noticed that the value of the potential of Ti/RuO₂ electrode short-circuited with Ti/TiO₂, $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$, is not stationary and decreases with time without illumination of Ti/TiO₂ photoelectrode. The extent and rapidity of this decrease was quite irregular, while the conditions of the experiment were formally the same. Such behavior can be attributed to corrosion of Ti/TiO₂ electrode in 0.5 M H₂SO₄, which most likely occurs at the bottom of the pores of anodically formed TiO₂ layer (see Fig. 1). Therefore, to avoid the interference of possible side reactions, which take place when Ti/TiO₂ and Ti/RuO₂ electrodes are short-circuited in acidic medium, the photoelectrochemical behavior of the system was investigated in alkaline medium, i.e., in the solution of 0.1 M KOH.

Photoelectrochemical investigations in 0.1 M KOH

The open-circuit potentials of Ti/TiO₂ and Ti/RuO₂ electrodes in 0.1 M KOH, similarly to acidic medium, were in the ranges 0.45–0.6 V and 0.76–0.87 V, respectively. However, contrary to the case discussed above, the value of $E_{i=0}$ of Ti/RuO₂ electrode remained practically the same after short-circuiting the electrode with Ti/TiO₂. Removal of dissolved oxygen from the solution also had no influence on the value of $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$. It was found, however, that the presence of dissolved oxygen influenced the photoelectrochemical processes of Ti/TiO₂ electrode.

The effect of UV irradiation on the $E_{i=0}$ of Ti/TiO₂ electrode in oxygen-free and oxygen-containing solution of 0.1 M KOH is shown in Fig. 4. One can see that upon

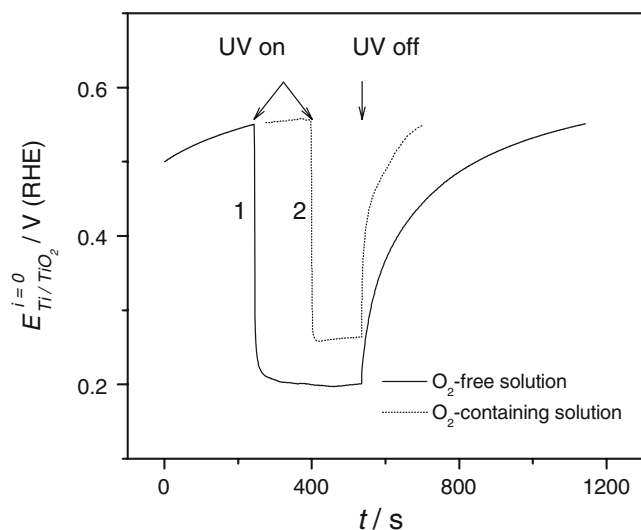


Fig. 4 Response of the open-circuit potential of Ti/TiO₂ electrode in oxygen-free (1) and oxygen-containing (2) solution of 0.1 M KOH to UV irradiation, $\lambda=365$ nm, $I_{\text{int}}=1$ mW cm⁻², 20 °C

irradiation the potential of Ti/TiO₂ in oxygen-free solution shifted instantly towards more negative values and stabilized at 0.2 V level (curve 1). Such negative shift of potential upon irradiation is typical for n-type semiconductor. It is important to note that such value of photopotential is not sufficient for H₂ evolution process to take place. It took about 10 min, i.e., a rather long period of time, for $E_{i=0}$ to regain its former value of 0.55 V after the UV irradiation was turned off. In oxygen-containing solution (Fig. 4, curve 2) $E_{i=0}$ stabilized at more positive value of 0.26 V and it took a considerably shorter period of time, i.e., about 3 min, for $E_{i=0}$ to regain its initial value after turning of the UV irradiation than it did in O₂-free solution. This means that oxygen dissolved in solution may act as additional acceptor of photogenerated electrons. Therefore, further experiments with Ti/RuO₂ electrode photocharging were performed in deaerated solutions. It is also important to note here that UV irradiation had no effect on the open-circuit potential of Ti/RuO₂ electrode.

The photoelectrochemical behavior of Ti/TiO₂ and Ti/RuO₂ electrodes connected through the 2 Ω resistance in O₂-free solution of 0.1 M KOH is shown in Fig. 5. One can see from curve 1 in Fig. 5a that $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$ decreases during UV illumination and within 30 min a quasi-stationary value of 0.26 V is reached. The most interesting observation is that, after UV illumination was turned off, $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$ did not spurt upwards like in the case of curve 1 in Fig. 4, but increased very slowly instead. During 30 min after turning off the UV light, $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$ increased by just 80 mV. Such behavior suggests that the photoelectrons generated in TiO₂ were effectively stored in Ti/RuO₂ electrode as reductive energy. The photocurrent, in turn, jumped to 80 μ A upon switching on the UV illumination (Fig. 5b). As $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$ approached 0.26 V, I_{ph} decreased continuously.

In order to find out what influence on the magnitude of I_{ph} has the starting value of $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$, Ti/RuO₂ electrode was polarized potentiostatically at 1.3 V for 5 min, then the cell was disconnected from the potentiostat, Ti/RuO₂ was connected with Ti/TiO₂ through the 2 Ω resistance, and UV illumination was turned on. The results are shown as curves 2 in Fig. 5a and b. One can see that the response of the system was quite similar to the one described before (curves 1, Fig. 5a, b). It took about 5 min longer for $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$ to reach the value of 0.26 V than in the case of curve 1 (Fig. 5a). The photocurrent jumped to 90 μ A at the moment of turning on the UV illumination. This value, however, is insignificantly higher than the one reached in curve 1 Fig. 5b, whereas the starting value of $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$ was significantly more positive, i.e., 1.28 V instead of 0.84 V (curves 1 and 2 in Fig. 5a). This means that I_{ph} has reached the limit, where the photon-to-electron conversion efficiency cannot be increased further to a greater extent under the conditions of experiment.

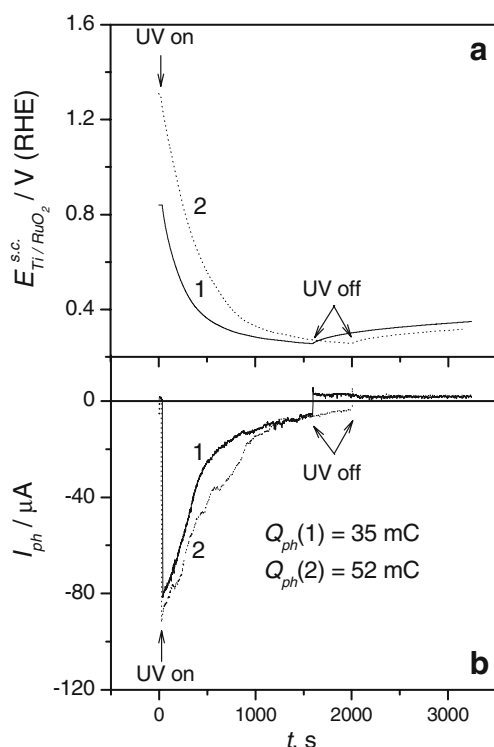


Fig. 5 Response of potential (a) and photocurrent (b) of Ti/TiO₂ and Ti/RuO₂ electrodes connected through the 2 Ω resistance to UV irradiation (λ=365 nm, I_{int}=1 mW cm⁻²) in the oxygen-free solution of 0.1 M KOH. Curves 1 and 2 were recorded starting from E^{s.c.}_{Ti/RuO₂} = 0.84V and 1.28 V, respectively

The amount of photogenerated charge— Q_{ph} , evaluated from integration of curves 1 and 2 in Fig. 5b, was ~35 and ~52 mC, respectively, which corresponds to a specific charge of ~21 and ~32 mAh g⁻¹, respectively. It was interesting to compare the value of Q_{ph} with the amount of anodic charge Q_a required for recovery of the potential of Ti/RuO₂ electrode from ~0.26 V to its initial value, i.e., 0.84 and 1.28 V (see Fig. 5a, curves 1 and 2). So the Ti/RuO₂ electrode was charged electrochemically by means of passing constant anodic current through the electrode till E attained the starting value of $E_{Ti/RuO_2}^{s.c.}$. Such method was employed in Refs. [2, 7]. The results of the experiment are presented in Fig. 6. The amount of charge required to drive the potential of Ti/RuO₂ electrode from ~0.26 V to 0.84 V and 1.28 V was 31 mC and 52 mC, respectively. One can see that the values of Q_a are close to those of Q_{ph} (Fig. 5b). It is also important to note here that the potential of Ti/RuO₂ electrode increased linearly during anodic charging (Fig. 6), what is typical for the charging of capacitor.

IPCE and mechanism of charge storage

The dependence of incident photon-to-electron conversion efficiency (IPCE) (Eq. 3) on the potential $E_{Ti/RuO_2}^{s.c.}$ of short-

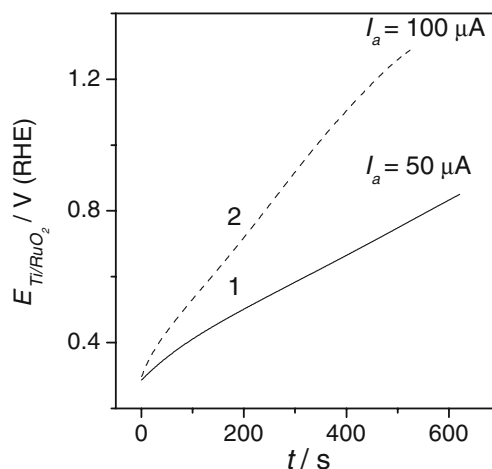


Fig. 6 Anodic charging of Ti/RuO₂ electrode from $E \approx 0.26$ V to initial state of 0.84 V (1) and 1.28 V (2) at 50 μA (1) and 100 μA (2) in oxygen-free solution of 0.1 M KOH; 20 °C

circuited Ti/TiO₂ and Ti/RuO₂ electrodes under UV illumination is shown in Fig. 7. One can see that, in the beginning, when $E_{Ti/RuO_2}^{s.c.}$ is at its uppermost values, the photon-to-electron conversion efficiency is very high, i.e., IPCE is 56% and 62% for the curves 1 and 2, respectively. However, as $E_{Ti/RuO_2}^{s.c.}$ approaches its lowermost quasi-stationary value of 0.26 V, the IPCE decreases practically to zero. The average photon-to-current conversion efficiency makes about 30%. It should be noted here that a similar value of IPCE (37.1%) was reported by Wang et. al in Ref. [2].

According to our recent study [22], it would follow that photoelectrons generated in TiO₂ are driven to Ti/RuO₂

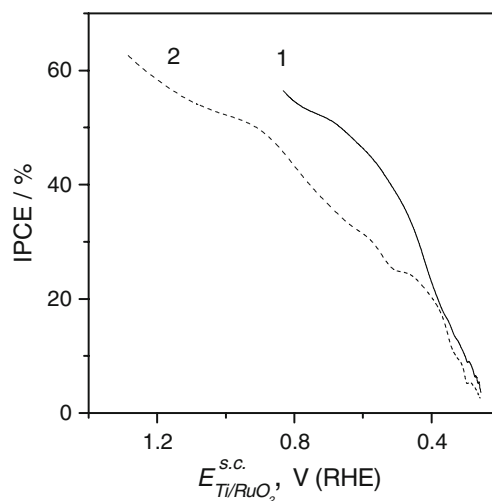
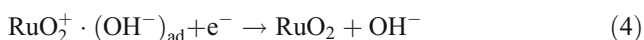


Fig. 7 Dependence of the photon-to-electron conversion efficiency IPCE (Eq. 3) on the potential of Ti/RuO₂ electrode connected to Ti/TiO₂ electrode through the 2 Ω resistance in the oxygen-free solution of 0.1 M KOH under UV illumination (λ=365 nm, I_{int}=1 mW cm⁻²); 20 °C. Curves 1 and 2 correspond to curves 1 and 2 in Fig. 5

electrode where they participate in the process of $\text{RuO}_2^+ \cdot (\text{OH}^-)_{\text{ad}}$ ionic pair reduction:



According to Eq. (4), the accumulation of negative charge within Ti/RuO₂ is compensated by dissociation of OH[−] ions from RuO₂⁺·(OH[−])_{ad} ionic pair. As $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$ becomes more negative (Fig. 5) and approaches the value of photopotential of Ti/TiO₂ electrode, i.e., 0.2 V (Fig. 4, curve 1), I_{ph} approaches zero value. In other words, as $E_{\text{Ti/RuO}_2}^{\text{s.c.}}$ decreases, the increasingly greater fraction of photogenerated charge carriers recombine instead of being separated, which is the reason of the decrease in photon-to-electron conversion efficiency shown in Fig. 7.

Holes, photogenerated in TiO₂, participate in oxidation of water. Formation of gas bubbles has been observed to take place on the surface of Ti/TiO₂ electrode during photoelectrochemical investigations. If the oxygen formed on Ti/TiO₂ electrode was not removed from the electrolyte, the electrochemical system under investigation would function like a photoelectrochemical cell, which transforms the energy of UV light into electricity, since the charge accumulated at Ti/RuO₂ electrode would be equivalently consumed for the reduction of photoelectrochemically formed oxygen.

Conclusions

It has been demonstrated that RuO₂ coating on titanium substrate effectively stores photoelectrons generated under UV irradiation in Ti/TiO₂ photoelectrode when these electrodes are short-circuited in deaerated solution of 0.1 M KOH. RuO₂ coating acts here as electrochemical capacitor, which accepts the photogenerated electrons. The intensity of photocurrent flowing from Ti/TiO₂ to Ti/RuO₂ electrode depends on the potential of the latter. Maximum value of photocurrent density was $\sim 180 \mu\text{A cm}^{-2}$, which corresponded to a maximum value of photon-to-electron conversion efficiency IPCE of about 60%. The amount of photogenerated charge Q_{ph} , which can be stored, depends on the capacitance of RuO₂ coating.

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