Supporting Information for

Quantifying Photocurrent Loss of a Single Particle-Particle Interface in Nanostructured Photoelectrodes

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1. Materials and methods

1.1 Materials and general methods

Chemicals and solvents were purchased commercially of typically analytical grade. A laboratory box furnace model LINDBERG Bluem (BF51842 Series) was used for the synthesis of TiO$_2$ nanorods. A Smart Coater 100 was used to spin cast TiO$_2$ nanorods on IDA-ITO electrodes. Transmission electron microscopy (TEM) was performed using EFI F20 TEM STEM. Scanning electron microscopy (SEM) was done on a Zeiss Gemini 500 at 1 keV.

Two major electrolyte solution conditions were used in this study (other conditions explored are described in Section 3.1 later) and both were purged by N$_2$ for 45 min:

1) In the absence of a hole scavenger: 0.1 M pH 8.3 sodium borate buffer, with 1 M sodium sulfate.
2) In the presence of a hole scavenger: the electrolyte solution above with addition of 0.6 M sodium sulfite.

1.2 Synthesis and characterization of TiO$_2$ nanorod

Rutile TiO$_2$ nanorods were prepared following the previously reported protocol.$^{1,2}$ Briefly, TiO$_2$ particles (ACROS Aerosol® P25, Cat. No. 384290500), sodium monophosphate (Mallinckrodt) and sodium chloride (MACRON) were mixed in 1:1:4 mass ratios and ground using a mortar and pestle. The fine mixture was transferred into a crucible and heated in a furnace at 850 °C for 12 h and then cooled down to room temperature. The mixture was dispersed in nanopure water (ELGA, 18 Ω), sonicated, and washed with hot water extensively. The purified rutile nanorods were collected via centrifugation and characterized using transmission and scanning electron microscopy. There nanorods are mainly single crystalline, ~200 nm in diameter and a few to ~10 μm in length (Figure S1D-I), with {100} facets along their sides and {011} facets at the ends$^2$ (Figure S1C).
Figure S1. TiO$_2$ nanorod characterization. (A) Representative TEM images of as-synthesized single-crystalline TiO$_2$ nanorods. (B, C) Higher resolution TEM image of a TiO$_2$ nanorod. (D-F) Length vs. diameter of a batch of 391 nanorods measured from TEM images (E), and histograms of their diameters (D) and lengths (F). (G-I) Same as D-F, but for the 46 nanorods whose photoelectrochemical behaviors were measured in this study from corresponding SEM images.

2. Bulk and sub-particle photoelectrochemical current measurements

2.1 Illustration of an interdigitated array (IDA) electrode

The commercially purchased IDA electrode is 20 mm long and 12 mm wide (Figure S2A), with 65 pairs of ITO strips, each ~11 μm in width, 100 ± 20 nm in thickness, and spaced apart every ~4 μm (Figure S2B-C).
2.2 Microfluidic photoelectrochemical cell and bulk/sub-particle photocurrent measurements

The cell has a 3-electrode configuration. To make the photoelectrochemical microfluidic cell, an interdigitated array (IDA) electrode without passivation membrane (ALS Co., Ltd., Japan) was used as the working electrode. 0.5 mm diameter Pt wire with 99.95% (metal basis; Alfa Aesar) purity was used as the counter electrode. RE-6 Ag/AgCl reference electrode (MW-2030) was purchased from BASi (Figure S3). Micro slides (plain, 1 × 3 in, 1.2 mm thick) were purchased from VWR. Micro Medical Tubing (Scientific Communication INC.) with 0.76 mm (I.D.) × 1.22 mm (O.D) was used to connect to the flow cell using Devcon (5 min Epoxy) transparent glue. The cell channel width is ~1 mm and height is ~100 μm, assembled using double-sided tapes (3M) sandwiched between the IDA-ITO electrode and a coverslip (Platinum Line®, 24×60). The outer edge of the photoelectrochemical microfluidic cell was sealed using LOCTITT, EA 10 (Henkel) glue. The cell was placed on an inverted microscope (Olympus IX71) and connected to the potentiostat (CH instrument model-1200A) for electrochemical measurements. A 375-nm laser (CrystaLaser DL375-020-O) was focused through a 60× NA1.2 water-immersion objective (Olympus UPlanApo) onto the cell. The same objective, along with a 1.6X magnification changer, was used to take
optical transmission images with an overall 96× magnification, giving a camera pixel size of ~167 nm. An EMCCD camera (ANDOR, iXon3; DU-897E-CS0-#BV) without EM gain was used to capture transmission images.

Electrolyte solution was flowed in the microfluidic cell using a 10 mL plastic syringe (BD 10 mL Luer-Lok™ Tip) and an automatic pump (Chemyx, model: Fusion 720) at a 25 μl/min flow rate. For the bulk measurement, a 0.7×0.7 cm² area that contains ~1000 nanorods were excited under epi illumination with the 375-nm laser. The light was chopped with 15 s on and 15 s off periods.

For sub-particle single-spot measurements, the focused 375-nm laser was parked at different positions on the TiO₂ nanorods and the corresponding photocurrent (in 0.02 s time resolution) was collected and extracted (see Section 2.4).

![Figure S3. Flow cell components. Schematic illustration of the microfluidic photoelectrochemical cell.](image)

### 2.3 Determination of the 375-nm laser focus size

The 375-nm laser was focused onto an ITO strip and a reflection image was captured. The bright laser spot could be easily seen (Figure S4A). The image of the focus spot was fitted with a 2-dimensional Gaussian function to obtain the centroid of the laser focus (Eq.1):

\[
I(x, y) = A \exp \left[ -\frac{(x-x_0)^2}{2\sigma_x^2} - \frac{(y-y_0)^2}{2\sigma_y^2} \right] + B
\]

Here \(I(x, y)\) is the intensity of the laser at position \((x, y)\), and \(A, B, (x_0, y_0), \) and \(\sigma_i\) are the amplitude, background, centroid, and standard deviation of the \(i\)-direction of the Gaussian function. The laser spot size was taken as the FWHM of this Gaussian function, which is about ~380 nm (Figure S4B).
2.4 Analysis of the current vs. time trace

Current vs. time traces were recorded using the three-electrode photoelectrochemical flow cell in the course of three consecutive 15 s on and 15 s off cycles of 375-nm laser illumination (Figure S5A) at any specified potential (stepped from -0.6 to +0.4 V). Electrochemical currents were collected at 0.02 s time resolution over 110 s time window (Figure S5A). Using a home-written MatLab program, the three pulses were first overlaid (Figure S5B) and then a linear background subtraction was applied using the signal before and after the light pulse (Figure S5D). Next, the current vs. time trace was smoothed by moving average of two consecutive points (blue trace, Figure S5D).

We assumed that the current jumps up when the light comes on. The histogram of the current show two peaks: the one at high current corresponds to the light on period while the other near zero corresponds to the light off period (Figure S5E). We set a threshold, which equals to center of the lower peak plus 1.9σ; σ is the standard deviation of light on or off peak in the histogram, whichever is larger. After thresholding the minimum index (i.e., time) among the data points that are larger than the threshold is set as the start of the pulse on period. By knowing the pulse width (i.e., 15 s), then we find the index of the point when the current drops. That is the light off point. Then, 10 data points (before light turns off) are averaged and this average was taken as the steady-state photocurrent (Figure S5F, G). A similar approach was performed to extract the photocurrent when the laser is parked at an adjacent position on quartz. Then, apparent photocurrent obtained from the quartz was subtracted out form TiO2 photocurrent to obtain net photocurrent (\(i_{ss}\)).

Figure S5. Current vs. time trace and photocurrent extraction. (A) Original current vs. time (i-t) trajectory over 3 on-off cycles at 0.4 V. (B) i-t trajectory after overlapping 3 cycles. (C, D) i-t background subtracted (C) and smoothed (D). (E) Histogram of current to identify on time (the yellow vertical bar indicates the threshold to find the start of light on period. (F) 10 data points
(indicated with green points) was chosen and averaged to extract corresponding steady-state photocurrent. (G) Expanded plot for clarification on panel F.

2.5 Determination of the distance between photocurrent measurement spot and ITO electrode edge

In both type-A and type-B photocurrent vs. distance measurements, the distance between laser spot and electrode edge was determined by using optical transmission images. Two images were captured. Image (1): laser was on and parked at the desired location (Figure S6A). Then, the laser was turned off and the optical transmission image with a 100 W halogen lamp (model: U-LH100L-3) was taken on the same location (Image (2), Figure S6B). Using Image (1), the \( x, y \) coordinates of the focused laser was obtained by fitting its intensity profile with a 2-dimentional Gaussian function, which includes a background offset (also see Section 2.3) (Eq. 1).

Next, Image (2) underwent a Matlab built-in edge detection algorithm with the Canny method to identify electrode and nanorods boundaries (Figure S6C). Then, the least squares regression method was applied to fit straight lines where electrode and nanorod meet or two nanorod meet (Figure S6D and E). Using the slopes and intercepts of the four lines, four \( (x, y) \) coordinates of the cross points were calculated (eq. 2, 3) (Figure S6C and D, red asterisks). The units of coordinates were later converted from camera pixels to nanometers (one pixel = 167.4 nm).

![Figure S6. Distance measurements between cross and electrode edge.](image)

For each cross point, its coordinates are related to the slopes and the intercepts of the two intersecting lines. For example:

\[
x_{\text{cor}}^{L_k} = -\frac{b_l - b_k}{m_l - m_k}
\]  

(2)
\[ y_{i,k}^{\text{for}} = m_i \left( \frac{b_i - b_k}{m_i - m_k} \right) + b_k \]  

(3)

Here \( m \) and \( b \) are the slope and intercept of each line of \( i, j, k, \) and \( l \) (Figure S6D, E).

The average of these four cross point coordinates (Figure S6D and E, red asterisk) was used to find the central location where the electrode edge and the nanorod crossed (Figure S6D, cyan asterisk) or the two nanorods crossed (Figure S6E, cyan asterisk).

For type-A spots, the distance between the laser spot and electrode edge was calculated from the separation of coordinates. For type-B spots, the distance is equal to the sum of the laser-to-cross distance and the cross-to-electrode-edge distance.

2.6 Calibration of distance measurements from optical images by those from SEM images

To estimate the error in the distance of laser spot position to the electrode edge measured from optical images, we measured the distance between the nanorod cross and the electrode edge in the optical images and compared the same distance measured from SEM images, which have higher resolution. Figure S7A shows the relationship between the measured distances using SEM and optical transmission images. Most data points fall on the diagonal line, demonstrating the reliability of the distance measurements from optical images. Figure S7B shows the ratio between distances measured from SEM and transmission images. The average ratio is 1.002 ± 0.005. Therefore, the difference is ~0.2% for an average distance of ~3.45 \( \mu \)m, corresponding to ~69 nm distance uncertainty. We further examined the absolute difference in the measured distance from SEM and optical transmission images. The average of their absolute difference is ~60 nm (Figure S7C); we have taken this value as the error in our distance measurements from optical transmission images.

Figure S7. SEM and optical transmission image distance measurement calibration. (A) Correlation plot of the measured distance between nanorod cross and electrode edge using SEM and optical transmission images. The blue line is a diagonal line. (B) Distribution of SEM and optical transmission images measured distance ratio. The average value is close to ~1. (C) Distribution of the absolute value of the difference between SEM and transmission images. The average value is ~60 nm.

3. Additional results and discussions

3.1 Waveguide effect is not significant in photocurrent measurements

Although nanorods can show waveguiding effects enabling light propagation along the length of the nanorods, we believe the waveguide effect is not significant under our experimental conditions for the following reasons:

(1) The material of waveguides needs to be transparent to the light that it guides to avoid loss. 3 In
our experiment, 375 nm laser light is absorbed strongly by the TiO₂ nanorods — it was chosen to be, so as to excite the charge carriers efficiently for photoelectrochemistry.

(2) Efficient waveguiding by the nanorod would cause the guided light emitting/emerging from the two ends of the nanorods. This emission was not observed in our experiment.

(3) The diameter of our nanorods is ~180 nm, significantly smaller than the wavelength of the 375 nm light, which would restrict light propagation along the length, leading to evanescent decay of light intensity away from the light entry (i.e., laser focus position) if waveguiding were in play. [Note the exact evanescent propagation properties need to be quantitatively evaluated (e.g., through simulations) taking into account nanorod shape, dielectric constant, and surrounding dielectric medium.]

(4) We have previously used the same experimental scheme of a focused 375 nm laser to measure single-spot photoelectrochemical currents on individual TiO₂ nanorods that laid flat and whose entire sides are in contact with the ITO substrate. There we observed that the single-spot photoelectrochemical current varied from spot to spot even within a single nanorod, indicating that the charge-carrier surface reactions are local to where the laser focus is. If the waveguide effect were to operate, the entire nanorod would be excited and the measured photoelectrochemical current should not differ significantly when the laser focus moved from one location to another within the same nanorod, which was not what we observed.

3.2 The local temperature of the spot on a TiO₂ nanorod illuminated by the focused laser beam is <1 degree higher than the temperature of the surrounding medium (i.e., room temperature)

We simulated the local temperature distribution as a function of time and the distance away from the illuminated spot based on the following the general heat conduction equation:

\[
\frac{\partial T}{\partial t} = \frac{k}{\rho C_p \rho} \left( \frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} \right) + \frac{H_v}{\rho C_p} \tag{4}
\]

where \( T \) is the temperature (K), \( t \) is the time (s), \( x, y \) are the spatial coordinates (m) (see Figure S8A), \( k \) is the thermal conductivity (= 8.65 W m⁻¹ K⁻¹ for TiO₂), \( \rho \) is the density (= 4260 kg m⁻³ for TiO₂), \( C_p \) is the specific heat capacity (= 697 J kg⁻¹ K⁻¹), and \( H_v \) is the heat generation term (W m⁻³). We can further simplify Eq. 4 by noting that the term \( \frac{\partial^2 T}{\partial x^2} \) and \( \frac{\partial^2 T}{\partial y^2} \) scale as \( \left( \frac{\Delta T}{(L_x)} \right)^2 \) and \( \left( \frac{\Delta T}{(L_y)} \right)^2 \), respectively, where \( L_x \) and \( L_y \) are the characteristic length scales of the nanorod in the \( x \) and \( y \) coordinates (about 180 and 4000 nm), respectively. Therefore, the term \( \frac{\partial^2 T}{\partial y^2} \) is \( (4000/180)^2 = 494 \) times smaller than the term \( \frac{\partial^2 T}{\partial x^2} \), which indicates that the former can be ignored. Therefore, Eq. 4 reduces to

\[
\frac{\partial T}{\partial t} = \frac{k}{\rho C_p \rho} \left( \frac{\partial^2 T}{\partial x^2} \right) + \frac{H_v}{\rho C_p} \tag{5}
\]

If we assume that all the light absorbed by a TiO₂ nanorod from a focused 375-nm laser beam (\( I_{375}, 0.056 \text{ MW cm}^{-2} \)) is converted to thermal energy (note this is an overestimation of the heating effect, which would result in an upper bound of the local temperature), and we know the absorption coefficient of TiO₂ (\( \alpha, 10^4 \text{ cm}^{-1} \) at 375 nm), the average nanorod diameter (\( h_{\text{avg}}, 180 \text{ nm} \)) and the illuminated area on the nanorod (\( A \)), the \( H_v \) term can be calculated as:

\[
H_v = \alpha h_{\text{avg}} I_{375} \frac{A}{h_{\text{avg}}} A = 10^4 \text{cm}^{-1} \times 0.056 \times 10^6 \text{W cm}^{-2} = 5.6 \times 10^{14} \text{ W m}^{-3}
\]
Note that here the $h_{avg}$ term and the $A$ term cancel out because $H_v$ is the amount of thermal energy generated per unit time per unit volume.

The initial condition here is that at time zero the temperature at any position on the TiO$_2$ nanorod is room temperature (298 K). The boundary condition is that at the nanorod/water interface, the temperature should be equal to the temperature of the surrounding medium (i.e., the constantly flowing aqueous electrolyte solution, which always has a temperature of 298 K). The boundary condition in this case is that at $x = 180/2$ nm = 90 nm (or −90 nm), the temperature should reach the local temperature of the water phase that is in contact with the nanorod. This temperature is room temperature (298 K), as discussed below.

In our experiments, the photoelectrochemical flow cell has a linear flow rate of 0.0042 m/s. The heat transport Peclet number ($Pe$) of the water phase, which quantifies the importance of heat convection relative to heat conduction in this fluid, can be calculated as:

$$Pe \equiv \frac{UL_{channel}}{k_w} = \frac{0.0042 \times 0.1}{0.58} = 3 \times 10^3$$

where $U$ is the linear flow rate (m/s), $L_{channel}$ is the length scale of our flow cell chamber in the flow direction (~0.1 m), $k_w$ is the thermal conductivity of water (0.58 W m$^{-1}$ K$^{-1}$), $\rho_w$ is the density of water (1000 kg m$^{-3}$), $C_{p,w}$ is the specific heat capacity of water (4180 J kg$^{-1}$ K$^{-1}$). The $Pe$ number is much greater than 1, indicating that in the fluid phase, the heat convection velocity is much greater than the heat conduction velocity. Therefore, any location in the water phase always has a temperature that equals the one at the reservoir (i.e., the container that supplies the electrolyte solution to the flow cell). That is, the local water temperature on the nanorod surface is always at room temperature.

Using Eq. 5 and the boundary condition discussed above, the temperature distribution as a function of $x$ and $t$ can be calculated, and is shown Figure S8B, from which it can be seen that the temperature approaches its steady state value quickly (i.e., at a time scale of ~10 ns). Figure S8C displays the steady state (i.e., $t = 100$ ns) temperature distribution versus $x$, and it can be clearly seen that the temperature at $x = 0$ (i.e., a point inside the nanorod that is the farthest away from the nanorod/water interface) is only slightly higher than the temperature of the surrounding solution by about 0.26 K. It is noteworthy that this temperature is the upper limit, and hence in our experiments this temperature rise should be less than 1 K.
Figure S8. Estimation of the local temperature distribution of the spot on a TiO2 nanorod illuminated by the focused laser beam. (A) Schematic illustration of the spatial coordinates $(x, y)$ used in the simulation of the temperature distribution, and the geometry of a representative nanorod. (B) Temperature distribution versus $x$ and $t$ for 100 ns, to show the steady state behavior. (C) The steady state temperature profile (i.e., $t = 100$ ns) as a function of $x$.

3.3 Survey of different electrolyte compositions and scavenger concentration

Figure S9. Survey of different electrolyte compositions on the photocurrent magnitude at the ensemble level: sodium borate buffer (0.1 M, pH: 8.3), KCl (1 M), Na2SO3 (0.2M) (green square), sodium borate buffer (0.1 M, pH: 8.3), Na2SO4 (1 M), Na2SO3 (0.2 M) (black circle), Na2SO4 (1 M, pH: 11), Na2SO3 (0.2 M) (red upward triangle), borate buffer (0.1 M), KCl (1 M) (pink downward triangle), sodium phosphate buffer (0.1 M, pH: 8.3), Na2SO4 (1 M), Na2SO3 (0.2 M) (blue diamond). The highest photocurrent obtained in sodium borate buffer (0.1 M, pH: 8.3), Na2SO4 (1 M) and Na2SO3 (0.2 M) electrolyte.
Figure S10. Photocurrent-vs-potential behavior at different scavenger concentrations at the bulk level. Electrolyte solution contains sodium borate buffer (0.1 M, pH 8.3), Na$_2$SO$_4$ (1 M), along with 0.2 M (green triangle), 0.6 M (black square), or 1 M Na$_2$SO$_3$ (red circle). The highest photocurrent magnitude at the positive potentials (e.g., 0.4 V) was obtained with 0.6 M Na$_2$SO$_3$.

3.4 Effect of hole scavenger on the onset potential vs. film thickness of TiO$_2$ particles

To further examine how the presence of hole scavenging affects the onset potential of $i$-$E$ behavior, we prepared a thick film of unshaped TiO$_2$ particle (ACROS Aerosol® P25, Cat. No. 384290500), a thick film of TiO$_2$ nanorods, and a monolayer film of TiO$_2$ nanorods on ITO as the photoanode and measured their steady-state photocurrent vs. the applied potential (Figure S11). These films were annealed for 1 h at 450°C before photoelectrochemistry measurements. Further details are described in Section 2.2.

Figure S11. Front illuminated bulk steady-state photocurrent versus potential measurement using different thicknesses of TiO$_2$ film on a ITO electrode substrate. (A) A thick film of spherical TiO$_2$ particles (ACROS Aerosol® P25). (B) A thick film of TiO$_2$ nanorods. (C) A monolayer of TiO$_2$ nanorod. The blue data points are taken in the presence of the hole scavenger and the red data points are in the absence of the hole scavenger ([$\text{SO}_3^{2-}$]=0.6 M).

It can be seen that thick films, either made of P25 particles or TiO$_2$ nanorods, show a negative shift in the onset potential and an increase in photocurrent upon adding the hole scavenger (Figure S11A, B), as observed for bulk electrodes in the presence of $\text{SO}_3^{2-}$. However, when a monolayer of TiO$_2$ nanorods on ITO was illuminated under similar conditions, a positive shift in the onset potential is observed (Figure S11C), similarly as we observed for single-spot measurements on individual TiO$_2$ nanorods (Figure 2B). The reason for this opposite shift in onset potential is unclear.
3.5 Additional examples of cross structures of TiO$_2$ nanorods on IDA ITO electrode

Figure S12. SEM images of more examples of crossed TiO$_2$ nanorods. The red and green circles indicate type-A and type-B photocurrent measurement spots.
3.6 Single-spot steady-state photocurrent versus distance to electrode edge at 0.3 V and 0.2 V in the presence of the hole scavenger

![Graph A](image1)

![Graph B](image2)

Figure S13. Photocurrent versus distance to electrode edge: for single-spot photocurrent measurements of TiO2 nanorods using a 375-nm laser focused onto type-A (red) and type-B (green) spots in the presence of the hole scavenger at (A) 0.3 V and (B) 0.2 V. The open circles indicate the binned (every 12 points) and averaged data. Error bars in x and y are s.d and s.e.m, respectively. The red and green solid lines are exponential fits (Eq. 1 in main text). The fitted parameters are summarized in Figure 3C-E.

3.7 Distribution of photocurrent losses among individual nanorod cross structures

Figure S14 below shows the distribution of steady-state photocurrent difference between the type-A and type-B spots from individual cross structures at 0.4 V in the presence of hole scavenger. The variation among individual cross structures is significant for many reasons: 1) the spots differ in distance to the ITO electrode edge among individual junctions; 2) the crosses/junctions differ in distance to the ITO electrode edge; 3) the TiO2 nanorods are known to have intra- and inter-nanorod heterogeneity in photoelectrochemical water oxidation performance, partly from surface heterogeneity of structural defects and impurity atoms, as we showed in our previous study.2
Figure S14. Histogram of steady-state photoelectrochemical current difference $\Delta i_{ss}$ between the type-A and type-B spots on 59 cross structures of TiO$_2$ nanorods at 0.4 V vs. Ag/AgCl in the presence of hole scavenger.

4. Additional references


(3) Deen, W. M. Analysis of Transport Phenomena; OUP USA, 2012.

