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Hou, Xuelan

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Active Area of Anodic TiO₂ Nanotube Arrays in Photo and Electrochemical Energy Storage Devices

Xuelan Hou*



active area, with respect to the Ti substrate shapes in different device applications is important in both academic research and commercialization due to the scale-up size effect.

KEYWORDS: active area, TiO₂ nanotube, anodic oxidation, Ti substrate shape, photo- and electrochemical energy storage

1. INTRODUCTION

Anodic TiO₂ nanotube (TNT) arrays have been intensively investigated and widely applied since its first preparation in 1984.¹ The applications are mainly in two sectors, solar energy utilization, for instance in photoelectrochemical water splitting (PECWS) cells,^{2,3} dye-sensitized solar cells (DSSCs),⁴ and PEC degradation cells,⁵ and electrichemical energy storage, such as in ion batteries,⁶ supercapacitors,⁷ and fuel cells.⁸

The current density is often employed to evaluate and compare the catalytic activity. To calculate the current density, the measure of the active area of the electrode has been necessary.⁹ Exactly, the ideal normalization of current should be performed with the real active area, viz., the number of the active sites. However, the measure of the real active surface of the catalyst is a challenge due to the complexity of the chemical reaction, reaction temperature, applied potential, etc., in (photo)electrochemical cells.9 Instead, three alternatives, geometric area (GA), electrochemically active surface area (ECSA), and Brunauer-Emmett-Teller (BET) area are often used for current density normalization, 9^{-12} even though there are drawbacks of describing the normalization of current according to one of the three mentioned active areas. With respect to anodic TiO₂, the definition of the active area becomes much more complex than the mentioned electrode due to the shape of the titanium (Ti) metal substrate.

The active areas of the catalysts on the Ti metal substrate are different from those one-sided active face substrates, such as transparent conductive oxide (TCO) glasses. The active areas of anodic TNT depend on the shapes of Ti metal substrates, viz., the exposing active faces, and the constructures of the application cells. Quite often, three sizes, Ti metal, TNT film, and the active area, need to be reported to specify the specimen.² For example, for the anodic TNT growth on a Ti foil, the film forms on both sides and edges; viz., identical twin films form on the two sides of one foil.³ Furthermore, Ti metal is flexible and can be made of different shapes, such as foil, wire, rod, foam, etc.¹³⁻¹⁶ Ti metal has been also coated on TCO glass (Ti/ TCO), and the composite Ti/TCO has been used in anodic oxidation. In this case, only one side grows TNT film. However, in general, the calculation of the active area for reporting the performance lacks clarity among the reports employing the anodic TNT apart from the three active areas: GA, ECSA, and BET.

Herein, we will particularly focus on the careful discussion of the three fundamental sizes: Ti metal, overall anodic TNT film, and active area of the TNT films in different devices to facilitate the data comparison.

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Figure 1. Scheme of the Ti substrate shape and sizes: (a) Ti/TCO, (b) Ti foil, and (c) Ti wire.

2. SIZE AND SHAPE

2.1. Ti Coated TCO Glass. In Figure 1a, Ti metal is coated on the conductive side of a piece of TCO glass, i.e., Ti/TCO.¹⁷ Only the "front face" grows TNTs in an electrochemical growth cell. The thickness of the coated Ti layer is often several hundred nanometers. The length and width of the Ti/TCO are often larger than the micrometer scale. The areas of films formed at the edges are negligible. Therefore, the active area in TNTs growth and in the electrochemical devices counts only the area of the front surface immersed in the electrolyte. The specific areas both in TNTs growth and applications do not arise unclearness in the active area calculations in this discussed topic. As a consequence, in the context of this article, the discussion will be focused on the foils and wires.

2.2. Ti Foil. The dimensions of Ti metal foil shown in Figure 1b have width, height, and thickness. For the size calculation of anodic TNT, the overall immersed area of the Ti foil in the anodic growth electrolyte needs to be summed up, including the areas of "front and back" faces and the edges. The edge surfaces are often negligible; for instance for a half-immersed foil with dimensions as $1 \text{ cm} \times 1 \text{ cm} \times 0.02 \text{ cm}$ foil, the area of the edges is 1/25 of that of both sides. The capillary effect of the immersed electrode in the growth cells and in a liquid-phase cell will be present in the discussion part. For the anodization process, both the areas of the front and back faces need to be reported. However, when TNT@Ti foil is used as a photoanode in a PEC WS cell, the dark current is negligible.¹⁸ In literature, the active area is often reported as the immersed front size facing the simulated 1-sun light source. In contrast, if the same sample is used as a cathode in an EC WS cell and a photocathode in a PECWS cell, the dark current becomes ineligible and the active surface should be summed up.^{3,19}

2.3. Ti Wire. Ti wire has measures of diameter (D) and length (L), as shown in Figure 1c. If the ratio of D/L is small, for instance, <100, the bottom film size of the cylinder is negligible for the anodization and most of the applications. Nonetheless, the error introduced due to neglecting the bottom size should be clear.

3. ACTIVE AREA IN PHOTOELECTRICAL DEVICES

3.1. Device as Electrode in Photoelectrochemical Water Splitting Cell. *3.1.1. Array as the Anode.* The anodic TNT arrays have been often used as the photoanodes in PECWS cells under 1-sun simulated light.^{20,21} For this case, the scheme in Figure 2 has been often illustrated in literature, showing one face of the TNT film, even though the specimen has two identical twin TNT films (Figure 3). This is reasonable because the dark current density of the TNT photoanode in a PECWS cell is often close to 0 mA/cm² and the Ti foil is non-transparent.¹⁸ Both the edge and the back side areas of the anodic TNT films are neglected in the calculation of the active



Figure 2. Scheme of the anodic TNT.



Figure 3. Scheme of the anodic TNT as photoanode in PECWS cell.

area. Rarely, there does exist an article that summed up the areas of both faces of the specimen in the calculation of the output of the PECWS cell, even though only one light source was in that test.²² Most of the published works on photoanode have only one face of the specimen exposed in the direct light irradiation as shown in Figure 3. However, the edges and the back side of the anodic TNT films are in the indirect irradiation; *viz.*, they live in the light scattering and radiation conditions. The contribution of the edges and back sides to the photocurrent density is hard to accurately quantify. Therefore, a systematic experiment and modeling with two light sources irradiating on both sides simultaneously, one light source and dark conditions, are needed to give a correct estimation.

3.1.2. Arrays as the Cathode. The anodic TNT arrays have been attractive as cathodes in electrochemical cells, such as in the nitrogen reduction reaction (NRR) to NH_3^{23} CO₂ reduction reaction (CO₂ RR) to carbon fuels,^{24,25} and hydrogen evolution reaction (HER).³ Assuming no capillary action, then all of the immersed surfaces should be summed up for performance calculation in such mentioned cases.

Anodic TNT arrays have been rarely investigated as the photocathode in the PECWS cell. Very recently, the photocathode from an anodic sample was prepared and applied in a PECWS cell, as in Figure 4. The modified anodic sample has an ineligible cathode performance in dark condition, achieving an overall current density of -100 mA in that work; one face anodic TNT film size is 1 cm^{2,3} That cathodic performance of anodic TNT is also supported in other conditions for NRR and CO₂ RR. Thus, the active area of the anodic TNT-based photo-



Figure 4. Scheme of anodic TNT as photocathode in PECWS cell. Reproduced with permission from ref 3. Copyright 2022 The Authors under a Creative Commons CC BY license, published by Elsevier.

cathode becomes much more complex than both cases in EC cells and as a photoanode in PECWS cells. All of the areas immerged in the electrolyte should be summed up, but with two kinds of different active areas, one facing to direct irradiation and another behind it, both contribute to the overall output. Therefore, with the photocathode in the PECWS cell, the overall current instead of current density is better for reporting the performance.

3.2. Hybrid cells as Electrode in Hybrid Cells. The hybrid cells are more complex, but the geometric area can be representative of the active area. Figure 5a shows a hybrid cell composed of a DSSC and a Li battery with anodic TNT as the anode for both half-cells.²⁶ The DSSC half-cell converts solar energy to electricity with one side of the Ti foil with a TNT array as the anode, while the Li battery uses the other TNT array as



Figure 5. (a) Design and principle of an integrated power pack of DSSC and Li battery based on double-sided anodic TNT films. Reprinted with permission from ref 26. Copyright 2012 American Chemical Society. (b) Structure schematic illustration and work mechanism of the PSC. (Dark-gray balls represent positive charges and light-gray balls represent electrons.) Reprinted with permission from ref 27. Copyright 2013 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim.

anode to store the electricity. The authors used "current density (mA/cm^2) " without mentioning the sizes of both the Ti foil and the TNT films. In this case, the sizes of the two array films are the same, and the geometric area is good for reporting the performance. Figure 5b illustrates another hybrid cell integrating a DSSC and a supercapacitor (SC) based on bipolar TNT arrays on Ti foils to form a stack-integrated photosupercapacitor (PSC).²⁷ The authors mentioned the dimension of the anodic TNT film is 2 cm × 2 cm, but in the cell, still many other components have different sizes, i.e., a piece of glass fiber separator 0.6 cm × 0.6 cm, a bipolar anodic TNT electrode 0.5 cm × 0.5 cm. The active areas of the DSSC, the SC, and the PSC are all given as 0.25 cm², respectively. It seems to be a reasonable way to evaluate the half-cells' performance and full-cell performance.

4. DISCUSSION

4.1. Capillary Effect. The anodic TNT electrode applied to the solid-state device and liquid phase involved device tells a different story due to the capillary effect. If the setup has liquid electrolytes, the capillary action will occur to result in an increase of the active area.⁹ The ignored increased active area because of the capillary effect will lead to a higher current density in the normalization. However, only a handful of cases pay attention to the capillary effect in their calculation in both PEC and EC cells for CO₂ reduction, water splitting, waste degradation, *etc.*^{9,28,29} Thus, for accurate calculation of the active area, minimizing the error introduced by the capillary effect is essential. A full immersion of the electrode is suggested to face this question.

4.2. How to Distinguish the Contribution of These Two Sides of the Electrode. As to the Ti foil substrate, the identical twin anodic TNT films are grown on both faces, Figure 4 and Figure 5a,b. Thus, distinguishing the contributions of these two sides of the electrode is an inevitable question. In electrochemical cells, the activity contributions of both sides are different due to the electric field distribution and ionic field effect. In the PEC cells, viz., under illumination, besides the question in EC cells, the light will introduce the photovoltage to the semiconductor, which will make the two sides much more complex. To solar-driven devices, two simulated light sources are desired for further testing, and also other cell designs are expected.

In literature, some prepared the one-sided TNT by sealing one face of the Ti foil with insulating tape.³⁰ Therefore, those will not need to face the discussed question. However, before the postannealing process, the insulating tapes are removed. Without using marks, there is an exposure of both the TNT film and the Ti foil during the electrochemical tests. Besides this new problem, other problems such as the effect of the different insulating glues and the possible reactions of the glue with the organic electrolyte may be introduced.

4.3. Active area. As mentioned in the Introduction, the real active area is impossible to measure. Thus, three alternatives, GA, ECSA, and BET areas, are often used. For nonilluminated applications, for example, electrochemical devices, ECSA is an option. However, to most solar-driven devices, both the contribution of the two sides and also the penetration of the light make it challenging. Ignore the inaccuracy and the reproducibility problem, the BET analysis is still not suggested for anodic TNT.¹¹ Thus, GA is used as a promising candidate due to being easy and direct. Other parameters such as the tube length or tube diameter can be assigned to other issues.

5. SUMMARY

Anodic TNT as arrays hold great potential as electrode materials in many photo- and electrochemical devices. It is important to clearly describe the active area sizes of the electrode in the applications to facilitate parallel comparisons among different materials in similar cells. It is sensible to describe clearly the three sizes, Ti metal, anodic TNT film, and active area, according to the shape and configuration of the electrode in the cell. A full immersion of the electrode is suggested for liquid phase involved cells. Geometric area is still suggested to be used as a promising candidate for the anodic TNT electrode due to its being easy and direct.

AUTHOR INFORMATION

Corresponding Author

Xuelan Hou – New Energy Technologies Group, Department of Applied Physics, School of Science, Aalto University, FI-00076 Aalto, Finland; ◎ orcid.org/0000-0001-9546-2643; Email: xuelan.hou@aalto.fi

Complete contact information is available at: https://pubs.acs.org/10.1021/acsaem.2c02492

Notes

The author declares no competing financial interest.

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