Nanometer TiO₂ Film-Based Solar Thin Film Manufacturing Technology and Performance Research

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1. Introduction

Energy is the basis for the survival and development of human society, and with the increasing development of society, people’s demand for energy has also shown an explosive growth trend, which has brought about energy shortages, ecological and environmental pollution, and other problems that are becoming increasingly prominent [1–4]. At present, the world’s energy consumption is still dominated by nonrenewable fossil energy, which is facing near depletion with continuous consumption. In addition, the exhaust gas produced during the use of fossil fuels also has a negative impact on the human environment [5]. In recent years, the frequency of haze in China has gradually increased and the scope of occurrence has gradually expanded, causing a great impact on people’s lives and health and social activities. Therefore, the development of new energy sources and the gradual change to a multi-energy structure is the current direction of human development [6, 7].

At present, human research and development of more extensive new energy sources mainly includes solar energy, water energy, nuclear energy, wind energy, hydrogen energy, biomass energy, and geothermal energy. Among them, solar energy, as the representative of clean energy, in other forms of energy, has incomparable advantages [8–10]. Firstly, solar energy reserves are extremely abundant, and since its life span can be maintained for another 10 billion years, solar energy can be regarded as an inexhaustible and permanent source of energy; secondly, there is no transportation problem, and it is widely distributed and easy to develop, even in remote mountainous areas or islands, etc. There is no geographical limitation; again, the energy received by the Earth from the sun each year is 30,000 times more than the total consumption of the world, so finally, solar energy is not discharged in the process of wastewater, waste gas, waste, etc [11].

The basic structure of DSSC mainly contains a transparent conductive oxide (TCO) substrate (generally FTO or ITO), a porous semiconductor oxide film, dye molecule, liquid electrolyte, and counter electrode composition. The main role of each part in the photoelectric conversion process is described as follows [12].

The conductive substrate plays the role of conducting carriers in the device. Usually, in order to improve the transmission performance of the carrier and the absorption efficiency of the device to sunlight, the conductive substrate is required to have a small square resistance and high light
transmission when preparing the device [13–15]. ITO is widely used in display panels because of its good conductivity, light transmission, and thin film thickness. However, in the process of preparing DSSCs, the semiconductor films are usually calcined at higher temperatures in order to enhance the bonding between nanoparticles and between the semiconductor films and the conductive substrate [16–18]. While the thermal stability of ITO is relatively poor, high temperatures will significantly increase the square resistance of ITO, which in turn affects the carrier transport efficiency [19].

As the core component of the entire cell device, the main role of the photoanode is to adsorb dye molecules, separate and transport carriers, and its microstructure (such as nanocrystalline type, film thickness, film porosity, flatness, and network structure) directly affects the amount of dye molecules adsorbed by the electrode, the absorption of incident light, and electron transport, which in turn directly affects the photovoltaic performance of the device [20, 21]. In the preparation process of DSSC, usually, a porous nano-oxide semiconductor film (e.g., TiO2, ZnO, or Al2O3) is deposited on a glass or polymer substrate with TCO [22].

For the photoanode, as a channel for electron transport, it is first necessary to have good electron transport capacity, which necessarily requires a good electrical connection between the semiconductor nanocrystals. On the other hand, as a carrier for adsorbed dyes, it is required that the film can adsorb as many dyes as possible in order to obtain a larger short-circuit current density [23]. In addition, the interior of the semiconductor should not be overly dense, and there should be some space to ensure good permeability of the liquid electrolyte inside it. In summary, the nanosemiconductor should have the following characteristics: (1) have a large specific surface area; the semiconductor film with a large specific surface area has a microscopic porous electrode structure, which is conducive to the adsorption of dye molecules when the electrode is sensitized and also conducive to the flow and permeability of the electrolyte, thus being able to generate a larger short-circuit current during operation; (2) the semiconductor oxide should have a high Fermi energy level: the open-circuit voltage of the DSSC is related to a high Fermi energy level, which is directly related to the Fermi energy level of the semiconductor oxide, and a high Fermi energy level is beneficial to the solar cell to obtain a high open-circuit voltage; (3) the energy level of the semiconductor material should be matched with other parts of the cell: the energy levels of the semiconductor oxide film, the dye molecule, and the oxidation-reduction electrolyte should be matched with each other, and a suitable energy level match can improve the efficiency of the dye generated electrons; and (4) fewer lattice defects and grain boundaries: lattice defects and crystal interfaces can trap carriers and make electron-hole complexes, thus reducing the photoelectric conversion efficiency of the cell.

2. Methodology of This Article

Dye molecules, as photoactive substances in DSSC, mainly assume the role of absorbing sunlight and generating excitons. Under the irradiation of sunlight, the dye molecule absorbs photons and is excited, and the electrons in the molecule jump from the HOMO energy level to the LOMO energy level, and then the excited dye molecule generates electrons, which are injected into the conduction band of the semiconductor oxide, while changing itself to the oxidation state, and the electrons injected into the conduction band flow out from the photoanode to achieve charge separation and generate photocurrent. Currently, the more common dyes used in DSSCs can be broadly classified as organic, inorganic, and complex [24].

The electrolyte has two main roles in DSSC: on the one hand, the electrolyte can rapidly conduct the holes generated by exciton separation. On the other hand, the reducing agent in the electrolyte must be able to rapidly reduce the dye in the oxidized state with its own reduction potential below the cell potential. The most common electrolytes are liquid, solid, and quasi-solid, among which the most common is the liquid electrolyte containing I7V redox electron pairs, and the cells prepared with it have high photoelectric conversion efficiency. However, liquid electrolytes also have obvious drawbacks [25–27].

1. It may easily lead to the desorption of the dye on the TiO2 surface, which may affect the life of the battery and its stability during operation.
2. The solvent in the electrolyte may react with the sensitized dye, thus leading to dye failure.
3. Liquid electrolyte leads to difficulty in sealing the device and results in poor performance of poorly sealed devices.
4. The volatility of the electrolyte solvent leads to cattie failure.
5. Low carrier mobility in the electrolyte, which is not conducive to carrier transport.

In response to these drawbacks, a rich variety of solid and quasi-solid electrolytes have also emerged to avoid the adverse effects of liquid electrolytes on DSSC devices.

Counter electrodes have two main roles in device operation: transporting holes and acting as catalysts for oxidation-reduction reactions in the electrolyte. At present, the common counter electrode is deposited on the conductive substrate by sputtering or pyrolysis. The platinum metal layer can reflect light to reduce the loss of incident light and improve the utilization of incident light in the cell, but can also play a catalytic role to increase the reduction rate of r. In addition to metallic platinum, other kinds of conductive films such as carbon films are also used in DSSC, but because of their poor catalytic effect, the counter electrodes used in the preparation of DSSC are still mainly uranium electrodes, as shown in Figure 1.

At present, nanocrystals are still the most ideal photoanode material. In terms of crystal type, TiO2 crystals have two types: crystalline (AnataSe) and rutile (RUTile), and amorphous (Brookite). The main difference between them is the different internal twisting and bonding in the T-octahedral structure, and these structural differences lead to different densities and electronic band structures of the two
crystalline forms. The density of anatase TiO$_2$ material is 3.895 g/cm$^3$ and the density of rutile TiO$_2$ material is 4.250 g/cm$^3$. The forbidden bandwidth of anatase TiO$_2$ is 3.2 eV, which is slightly larger than that of rutile 3.1 eV.

Due to the poor adsorption ability of rutile phase TiO$_2$ crystals to dye molecules, the specific surface area of nano films prepared from them is small, the electrons and holes in the films are easily compounded and the electron mobility in the films is low. Therefore, rutile phase TiO$_2$ is not suitable to be used as semiconductor oxides for the preparation of DSSC devices. In contrast, anatase TiO$_2$ films are suitable for the preparation of DSSC photoanode films because of their favorable dye adsorption, higher photoactivity, and higher electron mobility. However, anatase H$_2$O crystals undergo crystalline transformation to the rutile phase at high temperatures, and the critical temperature for the transformation is about 600 °C. Therefore, in order to obtain pure anatase phase TiO$_2$ films, the heat treatment temperature is usually controlled below 500 °C for the preparation of DSSC photoanode films. Besides, for other semiconductor oxides, the bonding strength between them and the dye molecules is lower than that between the dye molecules and TiO$_2$ molecules, and in DSSC, the weaker bonding is not conducive to the injection of photogenerated electrons from the dye molecules into the conduction band of the semiconductor oxide, and thus the photoelectric conversion efficiency of the device will greatly reduce the short-circuit current density and fill factor of the device, which in turn will reduce the photoelectric conversion efficiency of the device.

3. Experimental Method

3.1. Preparation of TiO$_2$ Sols. Add a certain amount of acetylacetone to 60 mL of anhydrous ethanol and stir magnetically for a few minutes. Then add a certain amount of butyl titanate and continue to stir magnetically for 30 min to obtain mixture 1. Take a certain amount of water to 50 mL of anhydrous ethanol, and adjust the pH from 3 to 4 with nitric acid to obtain mixture 2. Under vigorous magnetic stirring, add solution 2 to solution 1 slowly with a burette, and after the addition of drops, the mixture is stirred magnetically for 1 h and aged for 4 h to obtain a light yellow transparent TiO$_2$ sol. In this experiment, three groups of TiO$_2$ sols with different concentrations of precursors are made. The amounts of butyl titanate added are 34, 17, and 8.5 mL, respectively. The ratio of butyl titanate to water and acetylacetone was 1:2:1.

3.2. Preparation of TiO$_2$ Films. First, the ceramic pieces are cleaned by an ultrasonic wave and dried in the air. The ceramic sheets are coated by the dipping and pulling method. The pulling speed is 8 ~ 10 cm/min. After the coating operation, it is dried under natural conditions. Then, kept in an oven at 100 °C for 30 min, the above processes are repeated to obtain films of different thicknesses. In this experiment, ceramic sheets coated with three layers of TiO$_2$ films are used. Besides, the ceramic sheets coated in groups A, B, and C sols are recorded as A, B, and C, respectively.

3.3. XRD Testing. Due to the thin TiO$_2$ film plated on the ceramic sheet, the back and bottom of the ceramic sheet covered the TiO$_2$ diffraction peak during XRD detection, so the TiO$_2$ sol of groups A, B, and C was dried and treated in the...
same heat treatment manner as the film. The TiO₂ powder was analyzed by a dx-2000 X-ray diffractometer. The test conditions are Cu target, 35 kV, scanning speed of 2 (°)/min.

3.4. Performance Testing. The test is to put the ceramic sheet to be tested in the ultraviolet box (the main wavelength of the ultraviolet lamp is 254 nm and the illumination intensity is 2.5 MW/cm²) and measure the contact angle of water drops every 10 minutes. The test of photocatalytic degradation of methyl orange is to put three pieces of ceramic chips to be tested in a beaker containing 100 ml of methyl orange solution. The concentration of methyl orange is 5 mg/L. Put the beaker in the UV box, take methyl orange samples every 3 h for three times, and measure the absorbance of methyl orange samples with a Specord 200 UV-Vis spectrophotometer [31–33].

4. Results and Discussion

4.1. XRD Analysis Results. The XRD results of the three groups A, B, and C TiO₂ powders are shown in Figure 2.

The crystalline phases of A, B, and C powders are the same, and after comparing with the standard pattern of TiO₂ crystals, it is confirmed that all three groups of powders are anatase TiO₂, which indicates that TiO₂ has been completely crystallized into an anatase phase after calcination at 500 °C. The grain size of TiO₂ was calculated by Scherrer’s formula to be about 10 nm.

4.2. TiO₂ Hydrophilic Properties of Films. The contact angle of water droplets on the surface of three groups of ceramic sheets under UV irradiation TiO₂ was measured by [8]: the volume and diameter of the water droplets were measured and the contact angle was calculated according to Eq. \( \theta = \frac{4V}{\pi r³} \) (where \( r \) is the radius of the water droplet and \( V \) is the volume of the water droplet). The contact angle of three droplets was measured each time, and the average value was obtained. The hydrophilicity measurement results of the film are shown in Figure 3.

It can be seen from Figure 3 that the contact angle of water droplets on the surface of the film decreased sharply after 10 min of UV illumination and reached about 10°. With the extension of the illumination time, the contact angle of water droplets continued to decrease and stabilized after 30 min. It is reported in the literature [9] that when the contact angle \( \theta \) is less than 10°, the water droplets can completely infiltrate the surface and form a transparent water film, which will not scatter light and haze and has superhydrophilicity. It can be seen that the coated ceramics have superhydrophilic properties after 30 min of UV illumination. \( a, b, c \). There is no significant difference between the three groups of coated ceramics, which means that the precursor concentration has little effect on the hydrophilic properties of the film surface.

4.3. Photocatalytic Degradation Performance of TiO₂ Films on Methyl Orange. The photocatalytic decolorization reaction of methyl orange solution showed a quasi-level kinetic relationship, and the concentration of methyl orange solution was proportional to the absorbance: \( \frac{A₀}{A} = \ln \frac{C₀}{C} \). \( A₀ \) and \( C₀ \) were the concentrations of methyl orange solution at the beginning of light time and when the light time was \( t, v \), and \( A \) were the absorbance values of the solution at the beginning of light time and when the light time was \( t \), respectively. In this experiment, the degradation rate of methyl orange was expressed by \( \frac{(A₀ – A)}{A₀} \). \( a, b, \) and \( c \). The degradation rates of methyl orange for three groups of ceramic tablets are shown in Figure 4.

From Figure 4, it can be seen that the degradation rate of the films to methyl orange gradually increased with the increase of UV illumination time, which indicates that the nano-v films have some degradation to methyl orange. The degradation effect of the three groups of ceramic films \( a > b > c \), it can be seen that the degradation effect of the films
increases with the increase of the precursor concentration, which may be because the larger the concentration of the precursor, the larger the concentration of TiO₂ generated by its hydrolysis in the sol, and the more the amount of TiO₂ attached to the ceramic films during the coating, the better the effect of its photocatalytic degradation of methyl orange. However, if the concentration of TiO₂ in the sol is too large, the viscosity of the formed sol is too large, which is not conducive to the coating.

4.4. Effect of Heat Treatment Method on Film Properties.

The hydrophilic properties and photocatalytic degradation of methyl orange on the surface of the ceramic sheets a₁, b₁, and c₁ are shown in Figure 5. It can be seen from Figures 3 and 4 that the hydrophilicity and degradation performance of a₁, b₁, and c₁ are almost the same as those of a, b, and c in Figures 1 and 2 under UV illumination, Figure 6 indicating that the different heat treatments have little effect on the initial hydrophilicity and photocatalytic degradation of methyl orange.

The hydrophilic properties and photocatalytic degradation of methyl orange of the ceramic sheets a, b, and a₁, b₁ after being washed 100 times by QFS-A coating scrubbing tester are shown in Figure 7. The ceramic sheets a, b, a₁, b₁ after being washed are indicated by a’, a₁’, b’, and b₁’ respectively. Because the law of c, c₁ before and Figure 8 after washing is similar to that of a, a₁, b, and b₁, the graph of c, c₁ was not done.

It can be seen from Figure 7 that the hydrophilic energy of ceramic films a’, a₁’, b’, and b₁’ is worse than that of ceramic films a, b, a₁, and b₁. It means that the hydrophilic energy of ceramic films has decreased after washing. Such a phenomenon is caused by the surface structure change of the films with multiple washings and the attached TiO₂ partially shedding. However, the hydrophilic properties of a₁’ and b₁’ films decreased less compared with a₁, b₁. a’, b’ decreased more than others which indicates the surface structure of a₁’ and b₁’ films changed less. The adhesion of the films on the ceramic sheet is stronger, while the adhesion of a’ and b’ films is weaker. Because such adhesion is caused by the different heat treatment methods. Ceramic sheet a and b plating three layers of film calcination once, three layers of film thickness is larger, calcination of a large number of organic substances within the film volatilization and decomposition, and escape from the surface of the film, the film will appear a lot of internal pores, resulting in film fluffy and discontinuous, reducing the adhesion of the film on the substrate, while ceramic sheet a₁, b₁ plating a film
calcination once, a film thickness is smaller, calcination of the film volatilization and decomposition of organic material is also less, the film is more dense, the impact of recoating and calcination is less, and the film adhesion is stronger. In Figure 8, the degradation rate of the washed ceramic films $a_1'$ and $b_1'$ to methyl orange is greater than that of ceramic films $a'$ and $b'$ to methyl orange, which also shows that the adhesion of $a_1$, $b_1$ films is stronger than that of $a$, $b$ films.

5. Conclusions

The preparation of TiO$_2$ nanofilms has the advantages of immobilized catalysts, and simultaneously, its surface has photocatalytic activity and photophilic properties, which makes it valuable for theoretical studies and applications. The concentration of the sol-gel precursor had little effect on the hydrophilicity of the films, while the photocatalytic degradation of methyl orange increased with the increase in the precursor concentration. The film adhesion on the substrate and the stability of the film are improved by the calcination of each coating layer. In this work, the self-cleaning ceramics loaded with titanium dioxide nanofilms are prepared by the sol-gel method using ordinary ceramic sheets. Furthermore, the hydrophilic properties and methyl orange degradation properties of the films on the ceramic surface are tested, and the adhesion of TiO$_2$ films on the ceramic sheets under different heat treatments is also compared.

Data Availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.
Conflicts of Interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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