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Improving the performance of perovskite solar cells by electron beam evaporation processing

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Abstract: *Electron transport layer (ETL) plays an important role in the development of high performance perovskite solar cells (PSC). Here, we first compared the optical and surface morphology characteristics of titanium dioxide (TiO_2) thin films prepared by electron beam evaporation (EB) and hydrothermal (HT) method, as well as the device performance of perovskite solar cells, and then studied the effect of electron beam evaporation TiO_2 film thickness on the performance of perovskite solar cells. It was found that the morphology of TiO_2 ETL prepared by electron beam evaporation was more uniform and compact, and the surface root mean square roughness was lower than that of hydrothermal method, and the quality of the film was better. And the average transmittance of TiO_2 ETL prepared by electron beam evaporation was 94.67%. Compared with hydrothermal method, the PCE of TiO_2-based PSC devices based on electron beam evaporation was increased by 60%. The thickness of TiO_2 ETL was adjusted by electron beam evaporation, and a good surface morphology of the film was obtained. When the thickness was 75 nm, the ETL of the prepared PSC showed the best device performance, and the PCE reached 9.00%, showing excellent performance.*

Keywords: *TiO_2, electron transport layer, $CsPbI_{3-x}Br_x$ perovskite solar cell*

1. Introduction

The performance of organic-inorganic hybrid PSC is relatively high, but there are still major challenges in the stability of organic-inorganic hybrid PSC [1-3]. Chemical instability will occur at high temperature, but using Cs atoms instead of organic molecules as inorganic perovskite materials can solve this problem, because at high temperature, inorganic materials are usually more stable than organic materials. Titanium dioxide (TiO_2) has been widely used as the electron transport layer (ETL) of conventional PSC because of its high thermal stability, low cost and suitable energy level [4]. However, TiO_2 has several key inherent shortcomings, such as low electron mobility, high electron trap density and high catalytic activity, unexpected charge accumulation, recombination and instability under ultraviolet light, all of which lead to poor PCE, serious hysteresis and limited operation life [5-10].

Previously, Li et al. [11] prepared $CsPbIBr_2$ PSC using c-TiO_2 ETL and achieved 8.02% PCE. In the same year, J. Scott et al. [12] used solution method to prepare TiO_2 as inorganic $CsPbI_2Br$ PSC. The ETL, PCE reached 10.34%. Yu et al. [13] grew TiO_2 ETL by hydrothermal method and applied it to $CsPbIBr_2$ PSC, and the device efficiency reached 10.65%. Li et al. [14] reported that TiO_2 thin films were synthesized by solution method as ETL of $CsPbI_2Br$ PSC, and Spiro-MeOTAD was doped with Li salt. Perovskite solar cell devices can provide up to 12.6% efficiency. In the above study, TiO_2 ETL is prepared by solution method and hydrothermal method. Although these two preparation methods are simple and low cost, it is difficult to obtain uniform, large area and high quality TiO_2 films. Therefore, it is particularly important to explore a method to improve the uniformity and coverage of TiO_2 films. Electron beam evaporation is a relatively mature thin film preparation technology, which can prepare high quality thin films with uniform surface, and can be prepared in large area. Compared with hydrothermal method and solution method, there are relatively few studies on the application of TiO_2 films prepared by electron beam evaporation in $CsPbI_{3-x}Br_x$. More importantly, as far as we know, the mechanism of the difference in the performance of perovskite solar cells has not even been reported through the comparative study of TiO_2 films deposited by hydrothermal method and electron beam evaporation.

The exciton diffusion efficiency is the probability that the photogenerated exciton splits at the two-phase interface within the exciton lifetime [15, 16]. The general diffusion length of excitons is about 10

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nm, which is much smaller than the light absorption length, so the photoelectric conversion rate is increased. Therefore, the photoelectric conversion efficiency of organic photocells can be improved by optimizing the thickness of the electron transport layer. The particle size and thickness of the electron transport layer will have an impact on the anode. If the particle size distribution is uneven or the size is too large, the effect on the surface roughness at the bottom of the nanocrystalline film is not obvious, so the electrons cannot be extracted effectively [17]. The thickness of the designed electron transport layer is too large, from the point of view of simulating the incidence of sunlight, more incident light will be reflected, which will affect the absorption and utilization of light [18, 19]. Therefore, the design of electron transport layer with appropriate thickness is very important to improve the optoelectronic performance of perovskite solar cells.

In order to solve this problem, the electron transport layer was prepared by electron beam evaporation, and its process was explored. The TiO_2 thin films prepared by electron beam evaporation and hydrothermal method were compared, and the effects of different thickness of TiO_2 electron transport layer prepared by electron beam evaporation on the performance of perovskite solar cells were studied, and the mechanism of optimizing and improving the photovoltaic performance of perovskite solar cells by electron transport layer was revealed. Finally, an efficient TiO_2 base PSCs is obtained. The results show that the morphology of TiO_2 ETL prepared by electron beam evaporation is more uniform and compact, and the surface root mean square roughness is lower than that of hydrothermal method, and the quality of the film is better. And the average transmittance of TiO_2 ETL prepared by electron beam evaporation is 94.67%. Compared with hydrothermal method, the PCE of TiO_2-based PSC devices based on electron beam evaporation is increased by 60%. The thickness of TiO_2 ETL was adjusted by electron beam evaporation, and a good surface morphology of the film was obtained. When the thickness was 75 nm, the ETL of the prepared PSC showed the best device performance, and the PCE reached 9.00%, showing excellent performance.

2. Manuscript Preparation

TiO_2 thin films were deposited on fluorine-doped tin oxide-coated glass (FTO/glass) by EB evaporation. Before deposition, the chamber was evacuated to a base pressure of 5×10^{-3} Pa and the substrate was heated to 350 °C. TiO_2 particles were used as the source for evaporation, the deposition rate and film thickness were set at 0.5 Å·s^{-1} and 70 nm, and oxygen was introduced during the deposition process while the pressure was kept at 3.3×10^{-2} Pa. Annealing of the TiO2 thin film was carried out in oxygen at 500 °C for 2 h using a tube furnace. A mixture of 380.4 mg HPbI3, 187.08 mg CsI and 73.4 mg $PbBr_2$ was dissolved in DMF and DMSO and filtered with a 0.2 μm syringe filter to form $CsPbI_{3-x}Br_x$ solution. $CsPbI_{3-x}Br_x$ films were deposited by spin coating in glove box at 4,500 r.p.m. for 30 s and dried on a hot plate at 200 °C for 5 min. HTL was applied immediately after the perovskite film has cooled. The precursor solution for the HTL was prepared by dissolving 72.3 mg spiro-OMeTAD, 29 μl TBP and 18 μl Li-TFSI solution (520 mg Li-TFSI in 1 ml acetonitrile) in 1 ml CB. The HTL was deposited by spin coating at 7,050 r.p.m. for 30 s in glove box. The samples were then stored in a dark desiccator for the night. A 70-nm thick Ag layer was then deposited on top of the HTL through a metal shadow mask by thermal evaporation at a base pressure of about 2×10^{-3} Pa. The structure of the prepared device is shown in Figure 1.

Hydrothermal preparation of TiO_2 ETL: first of all, the deionized water is refrigerated in the refrigerator until it is completely reserved. In the process of preparation, the FTO glass cleaned and bombarded by plasma was fixed in a petri dish with high temperature tape, then 4.5 mL $TiCl_4$ was mixed with 200 mL ice water, and when the ice was nearly completely melted, it was introduced into the petri dish. Finally, the petri dish was placed in the oven at a modulation temperature of 70 °C for 50 minutes. The surface was washed with deionized water and dried with nitrogen, and then annealed at 200 °C for 30 minutes to obtain the TiO_2 electron transport layer.

The perovskite solar cells prepared by the two methods have the same structure, and the materials and preparation methods of other layers are also the same.

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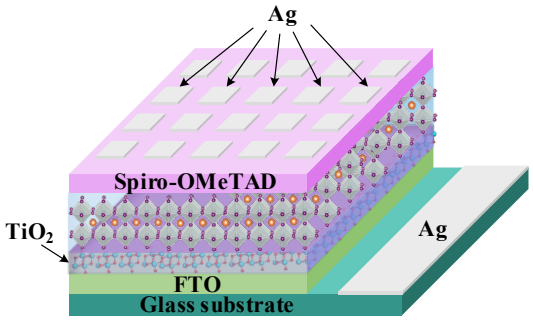

Figure 1: Structure diagram of $CsPbI_{3-x}Br_x$ perovskite solar cell.

3. Results and Discussion

3.1 Performance comparison of EB - TiO_2 and HT - TiO_2 ETL based on PSCs

The TiO_2 films prepared by electron beam evaporation and hydrothermal methods on FTO glass substrates were characterized by XRD diffraction, and the diffraction peaks were shown in figure 2, and compared with the TiO_2 films prepared on quartz glass. It is found that the diffraction angles of TiO_2 films prepared by the two methods are between 10 °and 70 °, and the peaks and positions are very similar. For TiO_2 films deposited on FTO, except for the diffraction peaks that belong to FTO itself, no new diffraction peaks are observed. For the TiO_2 films deposited on the glass substrate, only the amorphous drum of the glass substrate can be observed at about 23 °, which indicates that the prepared TiO_2 films are amorphous, which is usually due to the random arrangement of atoms in the volume [20]. The results show that there is no difference in crystal morphology between TiO_2 films prepared by electron beam evaporation and hydrothermal method on FTO substrate, and both are amorphous.

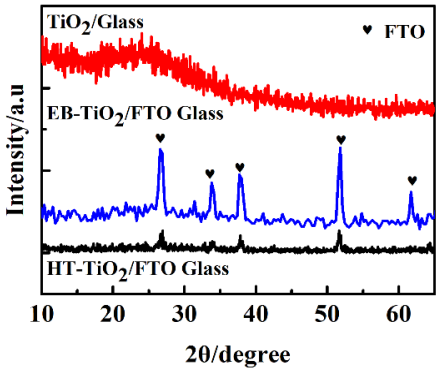

Figure 2: XRD diagram of TiO_2 thin films prepared by electron beam evaporation and hydrothermal Method

In addition to the crystal form of titanium dioxide, the morphology has an important influence on the light absorption, electron transport and electron trapping of the cell. To some extent, the crystallization quality of TiO_2 thin films can be evaluated by the grain size of perovskite and the roughness of crystal plane, so the surface morphology is characterized by SEM and AFM, as shown in figure 3. In order to study the mechanism, TiO_2 thin films with thickness of about 70 nm were prepared by electron beam evaporation and hydrothermal method, respectively. Figure 3 (a1) and (a2) are SEM diagrams of TiO_2 films prepared by two methods. It can be seen that the electron EB-TiO_2 film is relatively dense, the surface coverage is very high, close to the surface of FTO glass and the gap is relatively small. However, the density of the film prepared by HT-TiO_2 method is much lower than that of the film prepared by electron beam evaporation, with relatively dispersed particles and more voids. This may be related to the hydrothermal preparation process, which is through the reaction of $TiCl_4$ with ice water to form TiO_2, and then directly deposited on the FTO film, so the film is easily affected by the insufficient reaction of the solution to produce quality problems, while electron beam evaporation is controlled by rate, layer by layer to form a film, the film quality is relatively high. In a word, compared with HT-TiO_2 films, the morphology of EB-TiO_2 films is more uniform and compact, and the quality of EB-TiO_2 films is better.

Figure 3(b1) (b2) shows the AFM diagram of EB-TiO_2 and hydrothermal TiO_2 films. It can be seen

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from the figure that the root mean square roughness (RMS) of EB-TiO_2 film is 20.51 nm, while the RMS of HT-TiO_2 film is 87.96 nm, which is 3 times higher than that of EB-TiO_2 film, which also verifies the test results of SEM. The high surface roughness is also due to the formation of voids on the film surface due to the problem of preparation method. In summary, the RMS of TiO_2 thin films prepared by electron beam evaporation is lower and the surface is more uniform than that prepared by hydrothermal method.

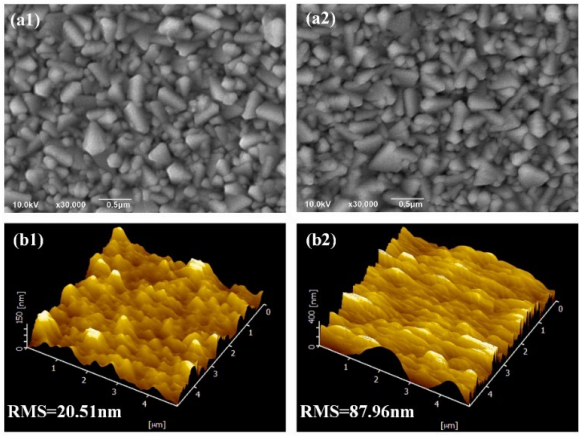

Figure 3: SEM and AFM diagrams of TiO_2 thin films by electron beam evaporation and hydrothermal method

Figure 4 shows the transmittance curves of FTO/TiO_2 films with a thickness of about 70 nm prepared by electron beam evaporation and hydrothermal methods. According to the calculation, the average transmittance of EB-TiO_2 and HT-TiO_2 is 94.67% and 92.31% respectively in the wavelength range of 380~800 nm. It can be seen that the light transmittance of EB-TiO_2 with the same thickness is better than that of HT-TiO_2, which is probably due to the excellent optical permeability and fewer interfacial voids brought by thin film interference theory (formula 1) and the high density of EB-TiO_2.

$$2nd + \frac{\lambda}{2} = k\lambda \quad (1)$$

k=1,2,3,4,5, d is the thickness of the film, n is the refractive index of TiO_2, the value is 2.55 ~ 2.76, and λ is the wavelength. When the formula (1) is satisfied, the interference of transmitted light is enhanced, which means that more light can penetrate the electron transport layer to reach the perovskite optical absorption layer, resulting in more photogenerated carriers and increasing the short-circuit current density, which is beneficial to improve the conversion efficiency of PSCs.

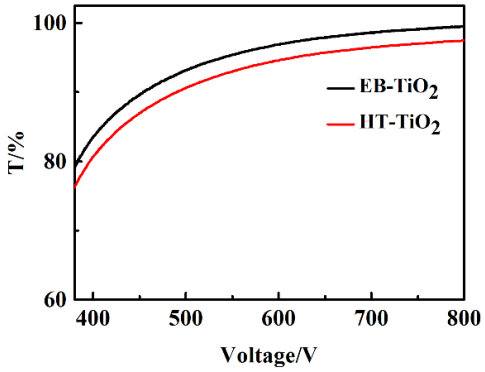

Figure 4: Transmission Spectra of TiO_2 Thin Films by Electron Beam Evaporation and Hydrothermal Method

Electron beam evaporation-TiO_2 and hydrothermal-TiO_2-based ETL were further made into complete PSCs. The J-V curve of the cell is shown in Figure 5, and the photoelectric performance parameters are shown in Table 1. The photoelectric performance parameters under the electron transport layer and the average efficiency of TiO_2 prepared by two methods are given in the table. It can be seen from the table that the devices prepared by electron beam evaporation-TiO_2 have higher short-circuit current density and higher open-circuit voltage than those prepared by hydrothermal-TiO_2 at the same thickness, and the filling factor and photoelectric conversion efficiency are also higher than that of electron beam evaporation-TiO_2. In contrast, the higher optical transmittance of electron beam evaporation-TiO_2 means

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that more photons can penetrate the electron transport layer to reach the perovskite absorption layer, resulting in an increase in the number of photogenerated carriers; at the same time, the high density of electron beam evaporation-TiO_2 films and the reduction of the number of holes also greatly reduce the defect density between the films, which are the advantages of electron beam evaporation. Although due to the immature preparation technology, the photoelectric conversion efficiency of electron beam evaporation-TiO_2-based PSCs prepared in this experiment has not exceeded that of hydrothermal-TiO_2-based PSCs, but it has lower short-circuit current density and higher open-circuit voltage, optical permeability and interfilm defect density are also obviously better than the latter. In addition, the TiO_2 dense layer used in planar perovskite solar cells with high open circuit voltage can be better prepared by electron beam evaporation. Because the electron beam evaporation method can be used to prepare large area battery components, high repetition rate of thin film preparation and adjustable industrial parameters, it has a very bright development prospect in the industrial production of perovskite solar cells in the future.

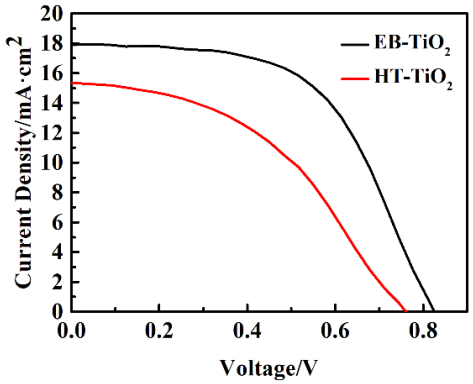

Figure 5: J-V curve of PSC prepared by electron beam evaporation and hydrothermal method TiO_2

Table 1: Photoelectric parameters of PSC of TiO_2 ETL prepared by electron beam evaporation and hydrothermal method

Preparation methods	V_{oc}/V	J_{sc}/mA·cm^{-2}	FF/%	PCE/%
EB-TiO_2	0.82	17.86	56	8.3
HT-TiO_2	0.76	15.35	44	5.12

3.2 Effect of thickness of TiO_2 thin Film by Electron Beam evaporation on the performance of PSCs Devices

We prepared four TiO_2 thin films with different thickness: 60 nm, 70 nm, 75 nm and 80 nm to study the effect of electron beam evaporation TiO_2 film thickness on the performance of PSCs devices. The solar simulator is used to provide light for perovskite solar cells, and the standard conditions for using solar cells are tested. During the test, the light is added first, and then the forward scanning voltage of-0.3 ~ 1.3 V is added to the solar cell. In this process, the current value is measured, and the data obtained are fitted by Origin software. The *J-V* curve is shown in figure 6. With the increase of film thickness, PCE increases at first and then decreases, which is attributed to the poor compactness of the prepared TiO_2 thin films. When the films are relatively thin, the surface grain spacing of nano-TiO_2 films is larger and there are many surface defects, which cannot effectively block the migration of holes from perovskite layer and HTL layer to FTO conductive glass, which will produce larger dark current and lower short-circuit current density of the cell [21].When the thickness of TiO_2 film increases, the TiO_2 layer can block the hole more effectively, but the electron transmission resistance increases accordingly, so that the short-circuit current increases at first and then decreases, and the changes of V_{oc} and FF with the thickness of TiO_2 film are not obvious. In this experiment, when the thickness of the film is 75 nm, the J_{sc} reaches the maximum, which is 13.36 mA/cm^2, and the PCE also reaches the maximum value, which is 9.00%.

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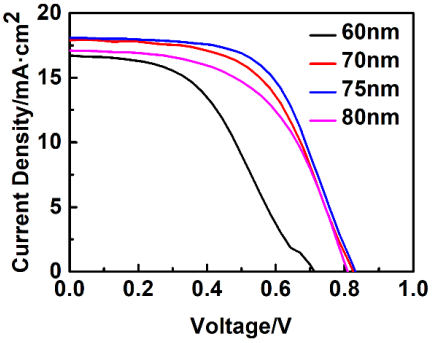

Figure 6: J-V curve of PSC prepared by TiO_2 with different thickness

Table 2: Photoelectric parameters of PSC with different thickness of TiO_2 ETL

Thickness /nm	V_{oc}/V	J_{sc}/mA·cm^{-2}	FF/%	R_s(Ω)	PCE/%
60	0.72	16.71	45	305.60	5.39
70	0.82	17.86	56	179.00	8.30
75	0.83	18.07	59	162.90	9.00
80	0.81	17.10	55	169.04	7.59

The series resistance of the device is also listed in Table 2. We find that the series resistance of the device decreases with the increase of the film thickness, reaching the minimum at 75 nm, and then increases with the increase of the film thickness, which indicates that the extraction ability of the electron transport layer is the strongest when the TiO_2 film is 75 nm. And in Table 2, the series resistance of 75 nm TiO_2 thin film devices is the lowest and the open circuit voltage is the highest. The efficiency of the device is the highest. It can be seen that the thickness of TiO_2 film has a great influence on the efficiency of the device.

When the film is too thin, there are too many defects in the film, which leads to the direct contact between the perovskite layer and FTO glass. Because the electron transport layer does not play a role, the resistance of free electrons entering the electrode directly through the perovskite film is greater, showing a larger series resistance, and a large number of defects in the TiO_2 film will also capture free electrons, which reduces the number of free electrons really collected by the electrode. In terms of parameters, the series resistance increases, while the short-circuit current, open-circuit voltage and other parameters are also sharply reduced, resulting in poor efficiency, so too thin TiO_2 layer is not a good choice.

When the TiO_2 is too thick, the first thing reflected in the parameters is the increase of series resistance. At the same time, due to the increase of thickness, the probability of radiation recombination increases when electrons are transmitted in the electron transport layer, which will eventually lead to a decrease in the number of electrons received by the electrode. After comparing the results, it is found that the perovskite solar cell device corresponding to the 75 nm thickness TiO_2 electron transport layer is the best. To sum up, the thickness of TiO_2 has a great influence on the performance of the device, so in the future research, paying attention to the influence of the thickness of each film on the device is a major research direction to improve the performance of the device.

4. Conclusions

In this paper, TiO_2 electron transport layer was prepared by electron beam evaporation and hydrothermal method. The effects of surface morphology, optics and perovskite solar cell performance of TiO_2 electron transport layer prepared by the two methods were studied. The results show that the electron transport layer of TiO_2 prepared by electron beam evaporation is more uniform and compact. And the film prepared by electron beam evaporation technology has a higher transmittance table by hydrothermal method, with an average transmittance of 94.67%. Therefore, the parameters of the device are improved by using the TiO_2 prepared based on electron beam evaporation in the electron transport layer of PSC, and the device PCE is 60% higher than that of the hydrothermal method. Then the effect of electron beam evaporation TiO_2 film thickness on the performance of perovskite solar cells was studied. The thickness of 75nm TiO_2 for PSC ETL has the best device performance.

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