

A Review of Methods for Preparing Thin Films of Titanium Oxide to Use in Dye-Sensitized Solar Cells (DSSCs)

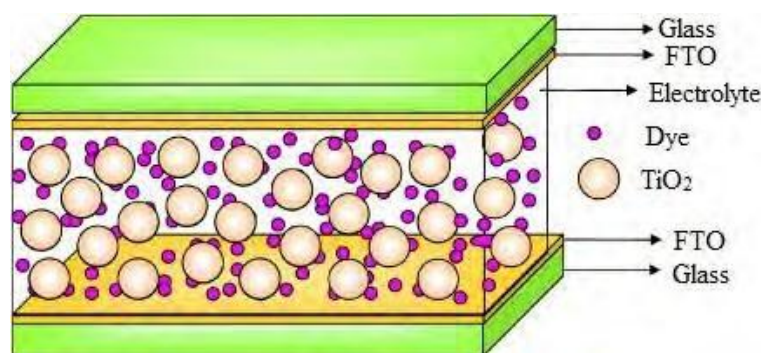
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ABSTRACT: In the last two decades, third-generation solar cells, i.e., dye-sensitized solar cells (DSSCs) or Gratzel cells, have become highly regarded due to their lower production costs and relatively high efficiency of converting light into electricity. One of the most important components of dye-sensitized solar cells is the titanium dioxide layer deposited on fluorine tin oxide (FTO), whose function is to accept the excited electrons of the dye and transfer them to the FTO substrate. Therefore, the preparation and application of this layer play an important role in the performance of dye-sensitized solar cells. The methods for developing and applying this layer are divided into two general classes: physical and chemical methods. Physical methods include laser pulses and spraying, and chemical methods include chemical vapor, sol-gel, simple coating, spin coating, spray pyrolysis coating, and electrocoating. Compared to physical methods, chemical methods are characterized by the fact that they are easy to perform and do not require packaged laboratory equipment and laboratory conditions. Among the chemical methods, the sol-gel method is superior to the other chemical methods because the thickness and properties of the produced coating can be controlled with ease and high accuracy. The aim of this study is to present methods for the fabrication of titanium dioxide thin films for dye-sensitized solar cells.

KEYWORDS: Solar cells, Photovoltaic properties, Titanium dioxide, Sol-gel, Laser pulse.

Graphical Abstract



1. INTRODUCTION

Introduction photovoltaic cells are used to convert direct sunlight into electricity and are used in the aerospace industry. Everyone is familiar with them in calculators and watches. In the last two decades, dye-sensitized solar cells (DSSCs) have received much attention due to their high and low cost compared to silicon solar cells [1]. The first dye-sensitized solar cell was developed by Gratzel et al. in 1991 [2]. The sample shown in **Figure 1** contains 5.

- 1) Dye usually made from the inorganic mineral complex ruthenium.
- 2) A porous oxide layer whose surface area should be high, and usually a layer of TiO_2 nanocrystals with a thickness of $10 \mu\text{m}$ is used.
- 3) Electrodes to collect electrons, usually a transparent conductive oxide layer. $\text{SnO}_2:\text{F}$.
- 4) The electrolyte solution, which must consist of a regenerative ion pair. For this purpose, a solution with an ion pair

(I^{-3}/T) is suitable.

5) A reducing electrode, which can be gold, platinum or graphite [3].

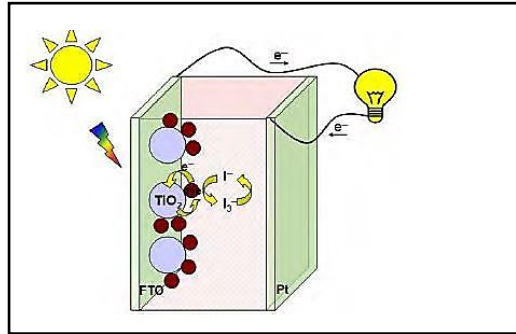


Figure 1: Dye-sensitized solar cell [4]

Energy conversion in a DSSCs is based on the transfer (injection) of an electron from the optically excited state of a dye-sensitizing conductor (base) of a crystallized semiconductor (TiO_2 is the most commonly used oxide semiconductor). These cells also use a liquid electrolyte (usually a redox active ether iodide pair). It is dissolved in an organic solvent that is used to deliver the electrons transferred to the titanium dioxide dye layer (in other words, it restores the ground state of the dye) [5]. This technology is now used all over the world, although in most countries it is a relatively new phenomenon that is not yet widely available to the public. **Figure 2** shows an example of a shopping center in Australia. On the roof of this center, solar panels are used to provide all the electrical energy needed [6]. One of the very new industrial applications of this technology is Sharp's production of cell phones and laptops. The company has installed a small solar cell on the back of the cell phone that can provide the electrical energy needed by exposing it to sunlight, even in cloudy and foggy conditions. The manufacture motorcycles and small optical vehicles and build solar cell charging stations used for these devices in the city are shown in **Figure 2** [7]. This technology seems to be able to replace many sources of electricity generation due to its rapid development. Since a large part of our country is located in desert areas and there is a large amount of solar energy, the industry needs to pay more attention to this clean energy.

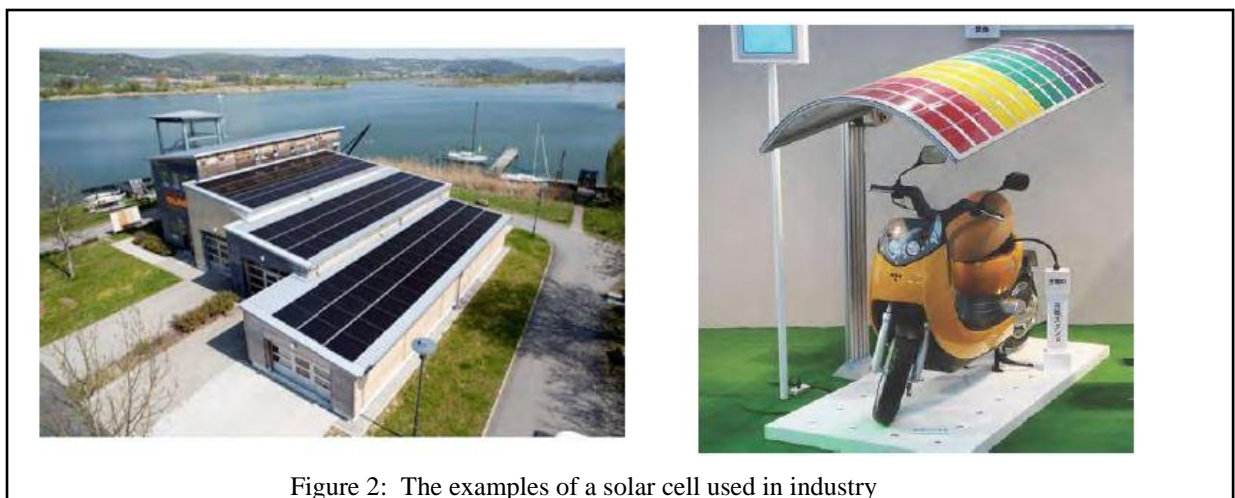


Figure 2: The examples of a solar cell used in industry

The purpose of this study is to present the methods of preparation and thin layer of titanium dioxide on the surface of FTO. It has been cited that the role of this exciting electron accepting layer is to color and play an important role in the function of the solar cells. The production of this layer is studied by two general chemical methods. The chemical methods are chemical vapor method, sol-gel method, immersion coating method, rotational coating method, thermal coating method and electrocoating method, and technical methods such as laser pulse method and spray method [8].

Chemical methods are more developed due to the lack of complex and practical laboratory equipment, and are paid more attention than research methods.

II. Chemical methods for the synthesis of nanoparticles and the preparation of thin layers of titanium dioxide

Two common physical and chemical methods are used for the synthesis of nanoparticles and the coating of titanium dioxide films, which are presented and the factors affecting each of them are explained.

A.. Physical methods for the preparation of nanoparticles and thin layers of titanium dioxide

A.1. Laser pulse method

A laser is a useful tool in industrial and laboratory applications, because it is narrow frequency bandwidth, coherence, and high power density. Various materials can be vaporized with a laser beam and then layered on a suitable substrate, this process is called laser pulse. This method is simple and only a few factors need to be controlled. Due to the very high heating of the target surface by the pulsed laser radiation, a stoichiometric layer is formed. This is the most important feature of this method. The advantages of this method are the stoichiometric transfer of the desired material (e.g. TiO_2) to the surface, the possibility of using inert or reactive gas in the environment, the possibility of using a wide range of temperatures and pressures, and the high variability in the choice of substrate. The process of the laser pulse method consists of first preparing a high-purity raw material and forming it from raw material plasma by irradiation of a laser pulse. The generated plasma is directed to the desired substrate at the appropriate temperature and the desired layer is formed on it. The disadvantage of this method is related to the high power of the laser, that it is not possible to produce samples with large areas, as voids, heterogeneity, pores and skin peeling may occur. Layer formation depends on important factors such as energy density, degree of ionization, type of raw material, temperature, and physicochemical properties of the substrate [9]. The main difference between this method and chemical methods is the possibility of forming multivalent titanium on the substrate. In the chemical method, no impurities occur due to the presence of oxygen molecules in the promoter itself or due to the application in an oxygen-rich environment. A thin film of titanium dioxide is prepared by the laser pulse method for using in photonic devices and, in particular, in solar cells that respond to dyes. For this purpose, various conditions for the application of this method, such as a wide range of pressure (from vacuum to 600 Torr), reaction temperature (from room temperature to 1273 K), and raw material (Ti and TiO_2) were investigated. The results showed that the titanium dioxide film prepared for using in the anode electrode of solar cells under the conditions of 100 Torr, 800 K ambient temperature, and TiO_2 as raw material had the best conversion efficiency (8%) [10].

A.2. Sputtering method

The steps for applying the sputtering method are 1) Formation of target ions: Gas atoms are thrown towards a target and the target ions are removed. For example, Ar ions are thrown at the raw material to remove its atoms. 2) Transfer to the substrate: After being removed, the target atoms must pass through sputtering gas (Ar) and plasma gas. Factors that reduce the efficiency are a random scattering of atoms, loss of energy of target atoms, and chemical reactions. The pressure of Ar gas is very important to form a suitable layer [11].

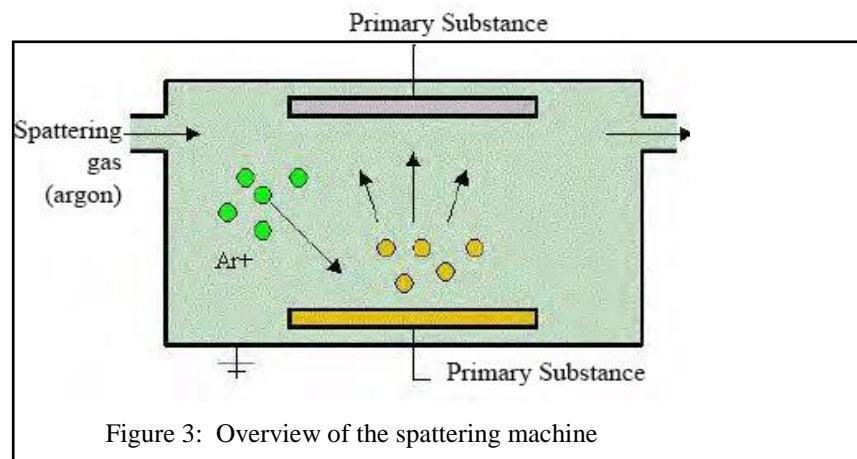


Figure 3: Overview of the sputtering machine

The advantages of this method include the high purity of the titanium dioxide layer produced, the uniformity of the layer obtained, and the possibility of combining several different nanomaterials. The disadvantages of this method are the dependence of the properties of the produced titanium dioxide layers on the angle coating and the pressure applied during the production process. The best conditions for producing a uniform film with suitable application in solar cells are a 50°-degree angle of the substrate and a pressure of 2 Pascal. The conversion efficiency of a solar cell made with such an electrode is 8% [12,13]. Of course, it should be noted that the strength of this method lies in the possibility of using two or more nanoparticles. For example, the use of nanoparticles of zinc oxide and titanium dioxide can be suitable electrode for using in solar cells. Using such an electrode (without further modifications), the maximum power is about 72% and the conversion efficiency will be 9.5% that it is increased [14].

III. Chemical methods for the synthesis of nanoparticles and the preparation of thin layers of titanium dioxide

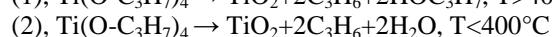
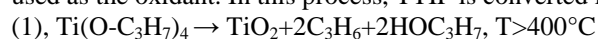
A. The chemical vapor deposition method

In chemical vapor deposition, the deposited material is passed through a propellant gas (e.g., SiH₄ and TiCl₄) into a reactive chamber. The propellant gas is evaporated from the substrate to cover the entire substrate at a high temperature (800-1000°C). After the molecules come into contact with the hot surface, the unstable and gaseous parts decompose and the parts that have the desired composition settle on the substrate. The advantage of coatings produced by the chemical vapor deposition method over methods is that a hard and abrasion resistant coating. Some of the disadvantages and problems of this method are:

1) A high temperature is required for the coating, so the materials used in this method must withstand this temperature, which reduces the selectivity of the material.

2) Depending on the type of promoter, the reactor chamber can become toxic and flammable.

Recently, the thin films of titanium dioxide by chemical vapor deposition with a metal organic precursor, Metal Organic Chemical Vapor Deposition (MOCVD) is well known [15],[16]. For making the anode electrode of solar cells, glass as the desired substrate and titanium isopropoxide (TTIP), which is liquid at room temperature (melting point 20°C), is used as the reaction promoter. The material is first vaporized in a glass capsule whose temperature is controlled by a hot plate device, and then returned to the reactor chamber with oxygen gas, as well as pure oxygen is used as the oxidant. In this process, TTIP is converted into TiO₂ according to reactions 1 and 2.



The fabricated solar cell with this electrode has an efficiency of 7% [17]. The best results for the fabrication of anode electrodes in solar cells were published by Nam et al. They consisted the promoter material Ti (OⁱPr)₄ and used a glass substrate at a temperature of 800 ° C and a solar cell with this electrode with an efficiency of 8.22% [18].

B. Sol-gel method

Chemical methods such as the chemical vapor deposition method are called dry chemical methods, as opposed to methods that use aqueous compounds or compounds that use hydroxyl and alkalis during the process, that they are called wet chemical. Thus, the sol-gel method is a wet chemical method. The advantages of this method over other methods are simpler synthesis steps, more homogeneous particle distribution and higher reactivity of chemical components, reduction of manufacturing steps, controllable particle size (nanoscale) and higher density. The preparation of a thin film by the sol-gel process is slightly different from the preparation of nano-powders and consists of 4 steps:

1) The colloidal particles are dispersed in a liquid to form a tubercle.

2) By spraying the solution, on the layers.

3) As the particles move on the surface and form bonds with each other and form a polymer or a stable gel

4) Upon final heating, the organic and inorganic compounds remaining in the gel form a crystalline or bicolor network (depending on the temperature applied). In the following, in this study, two reports on the preparation of titanium dioxide nanoparticles by the sol-gel method using additional thermal (hydrothermal) and sedimentary methods are brought. Chen et al. used the thermal method [19]. TiO₂ nanoparticles were prepared by hydrothermal deposition of titanium dioxide peroxide gel by hydrothermal or calcination methods. Some of the titanium isopropoxides were dissolved in ethanol without water and then mixed with an alcoholic aqueous solution with a molar ratio of water-oxide 170.3. The isopropoxide solution is gradually added to the aqueous solution at room temperature with vigorous stirring.

Using a centrifuge, remove the precipitate and re dissolve in alcohol to remove all debris. This washing procedure is repeated 5 times. The final precipitate is calcined in the presence of oxygen at 450 °C. Conversion efficiency in fabricated solar cells with this electrode. In another report, the study of Kang et al is considered [20]. For the preparation of nanoparticles, first dilute NH_4OH or HNO_3 solution with water to achieve the concentration of 2.5% NH_4OH . Then and $(\text{Ti}(\text{OC}_4\text{H}_9)_4)$, dilute NH_4OH with HNO_3 and add it gradually with stirring to obtain a clear solution. The hydrolysate product is a precipitation that breaks the product at a temperature of 40-50 °C. It is dried and then calcined. The conversion efficiency in solar cells made with this electrode is 4.8%. In another report, the effect of solvent and temperature on the produced electrode was studied and it was found that the highest conversion efficiency for a solar cell is obtained when the sol-gel process takes place at a temperature of 450 °C and an aqueous-alcoholic solution with a ratio of 1:3 M, improving the conversion efficiency of the solar cell by about 1.3%.

C. Dip coating method

In this method, a sol-gel solution with a specific concentration and viscosity is prepared. Then a suitable substrate is dipped into the solution at a certain rate and, after being suspended in the solution is removed. The thickness of the coating is mainly determined on the substrate emerges from the solid contents and the viscosity of the solution. The interesting thing about this method is choosing the right viscosity, the thickness of the coating can be changed from 20 nm to 50 nm. After coating, the prepared gel layer on the substrate is heated so that only the desired material remains and the other components are evaporated. The advantage of this method is that each layer can be recoated after heating. The thickness of the layer, as well as other properties such as transparency and porosity, depend on the number of dipping operations. The thin films of titanium dioxide nanostructures is fabricated in Ref [21]. Titanium isopropoxide, isopropyl and chloric acids were used to prepare the initial solution. The stirring solution was stirred at room temperature for 1 hour and then allowed to stand for 4 hours. The viscosity can be adjusted to the desired value. The substrate is cleaned with disinfectants (acetone and distilled water) to ensure uniform coating. The substrate is immersed vertically in the prepared solution bath and pulled out at a speed of 1 mm/s. The substrate is then removed from the solution bath. This is the required speed for the coating to condense and hydrate on the substrate. During the immersion process, the coating forms on both sides of the substrate. The coatings are air dried for 30 minutes form chemical bonds between the coating and the substrate. They are then heated to 100 –400 °C for 10 minutes as mentioned. The thickness of the prepared layer on the substrate depends on the rate at which it emerges from solution, and the thickness has a significant impact on the performance of the solar cell. For example, two solar cells with anode electrode thickness of 10 and 15 μm have an efficiency from 1.68 to 2.40%, respectively. Therefore, it is necessary to choose the best time according to the substrate material and environmental conditions, which is the optimal speed for the glass surface at a temperature of 25 °C [22] complete crystallization of the target province on the substrate [23].

D. Spin coating method

In this method, the solution is first prepared by the sol-gel method. It is then placed on a substrate that rotates perpendicular to the axis at a certain rate to achieve a certain thickness. By rotating around the axis, the solution is gradually spread on the surface of the substrate and forms a thin layer on the surface. At the same time, the substrate is heated to the appropriate temperature to form a crystalline layer, and the solvent evaporates (Figure 4 and 5). The thickness of such coatings ranges from 10 nm to 10 μm . The quality of the coating depends on the rheological properties of the coating liquid.

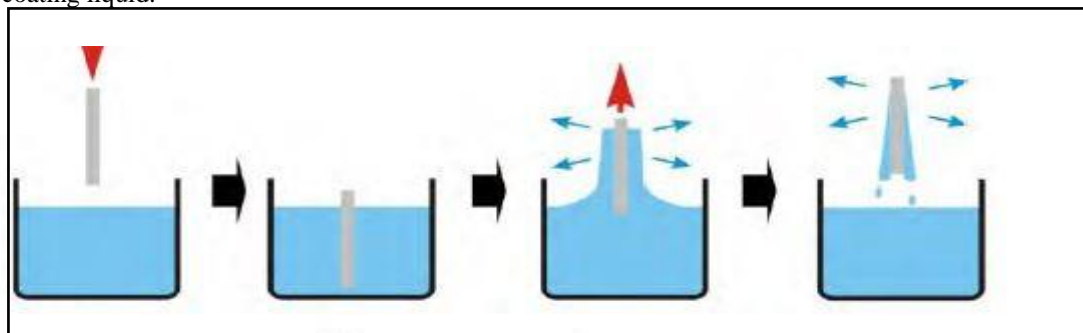


Figure 4: Submerged cover process

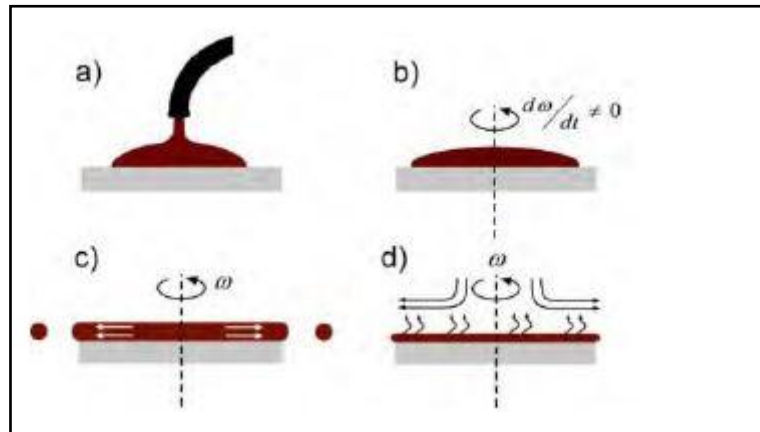


Figure 5: Rotational coating process

The description of the preparation thin films of titanium dioxide nanostructures is reported in this way. Titanium isopropoxide is commonly used as a promoter to prepare the desired solution. In this method, titanium tributoxide ($\text{Ti}(\text{O}i\text{Bu})_3$) was used as a promoter. The rotation speed of the device is 3000 rpm for 40 s and then it remains at a temperature of 150°C for 10 min on the hot plate to prepare the next layer to thicken the layer [24]. The advantages of this method are the elimination or integration of rotational variables, the ability to achieve different thicknesses by changing the spin speed or the viscosity of the solution, the ability to produce a uniform film, low cost, and a fast application procedure. This method has only one major drawback, which is that it is not possible to coat large substrates at high velocity, because the produced coating is not uniform. Therefore, for large parts, the coating speed must be low and controlled. An important application of this method is the fabrication of anode electrodes for using in solar cells. For example, Wang et al. used this method to fabricate solar cells with an efficiency of 25.3%, and another report using this method presented solar cells with an efficiency of 8.7% [25]. According to Figure 6, this tank is connected to the nozzle by a silicone tube. The nozzle has a diameter of about 0.1 mm to crush the particles and spray them from a certain height, which can be adjusted, onto a hot plate on which placed the substrates for coating. Gas is used to deliver the solution into the nozzle and spray onto the substrates, which affects the gas pressure and nozzle diameter, particle size and coating quality. Since the substrate is placed on a hot plate at a temperature suitable for the chemical reaction of the solution, after the solution is sprayed from the nozzle onto the substrate with gas pressure, the excess material and impurities are immediately evaporated and only the material desired (TiO_2) remains on the substrate to form the coating. After the completed coating, the substrate is cooled to ambient temperature. In order to form an even and homogeneous layer on the substrate, the nozzle performs a pivoting motion similar to the spray method and the heating plate performs a rotating motion with adjustable speed. The installation of a hood helps to remove the reaction gasses on the surface of the substrate and prevents them from being disturbed when spraying the solution onto the bed (Figure 6). The advantages of this method are the ability to prepare large surfaces with different thicknesses, the ability to produce a monotonic film, and the low price. The main disadvantage of this method is that the temperature must be controlled to achieve a suitable film thickness. Otherwise, the produced films have irregularities and undesirable pores [24]. An important application of this method is the fabrication of anode electrodes for using in solar cells. For example, in one study, solar cells with a conversion efficiency of 8.4% were fabricated with using this method [25] and in another report, a solar cell with a efficiency of 8.7% was presented [26].

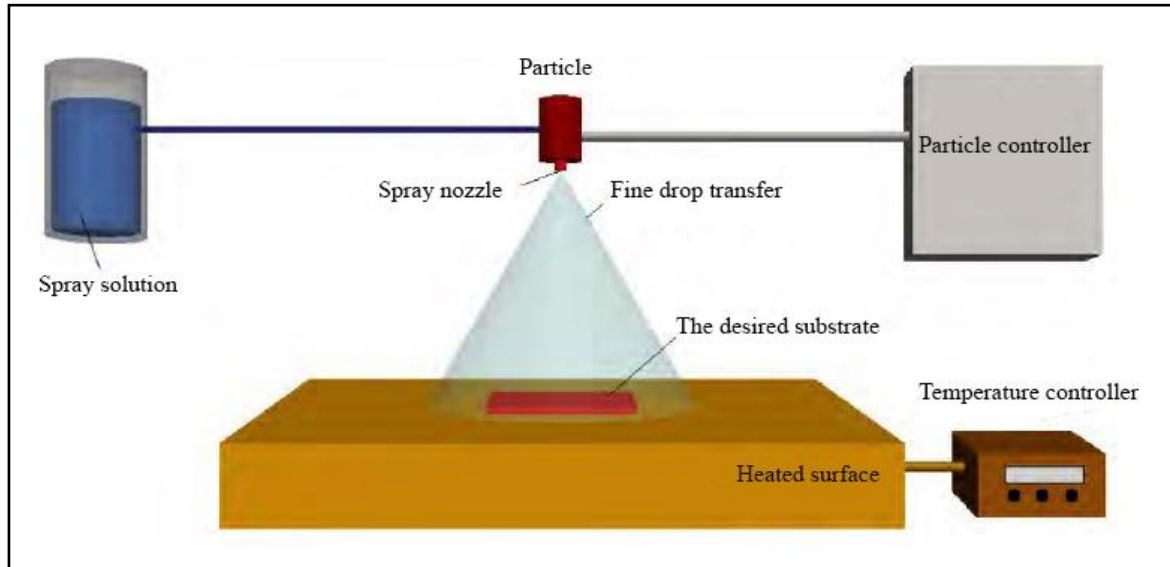


Figure 6: Thermal diffusion coating layer process

growth and uniformity of the film. The advantages of this method are the production of a very uniform coating without cracks and pores, the production of a high-purity coating, the application to a wide range of materials (metals, ceramics, polymers, etc.), and the easy control of the coating composition and low price [27]. An important application of this method is the fabrication of anode electrodes for using in solar cells. For example, solar cells with a conversion efficiency of 3.6% were fabricated in the study with using this method [28], and a solar cell with an efficiency of 2.8% was presented in another report Ref [28].

IV. CONCLUSION

In the last two decades, the use of solar energy to generate electricity has received much attention as a biocompatible source. Solar cells are used to convert solar energy into electrical energy. One of the main components of solar cells is the working electrode, whose function is to accept the excited electrons of the dye and transfer them to the FTO surface. There are two general classes of physical and chemical methods for fabricating anode electrodes in solar cells. The physical methods of bartending are: laser pulse and spraying, and the chemical methods include chemical vapor, sol-gel, immersion, rotational, thermal spray, and electrocoating. In the laser pulse method, it is possible to choose a wide substrate and stoichiometric transfer of the word material, but the disadvantage of this method is the formation of pores and defects in the coating, which does not exist in the spray method. To produce a hard and wear-resistant coating, it is necessary to use the chemical vapor method. One of the most important methods, which has fewer and simpler manufacturing steps and a homogeneous particle size distribution, is the sol-gel method, which belongs to the category of more chemical methods. If the desired thickness is not achieved, the dip coating method can be used for recoating. The rotational coating method can be used to achieve coatings of various thicknesses with small variable parameters. To coat large areas with different thicknesses, the thermal spray method must be used, but the temperature must be carefully controlled to achieve a uniform coating. Electrocoating method is very important because it produces uniform coating with high purity, low price and possibility of application for different levels. Chemical methods are generally less complex than physical methods because they are facilitated to apply and require less complicated laboratory equipment and conditions. Among chemical methods, the sol-gel method is superior due to the ability to control the properties and thickness of the produced layer with great ease and accurate.

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