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POLYTECHNIC UNIVERSITY
香港理工大學

Department of Applied Physics

Final Year Project Report

Project Title

Water Purification: Material Study

**Fabrication of hexagonally-ordered bowl-shaped
and micro-cavity TiO₂ nanostructures by
microsphere lithography**

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Abstract

This report aims to present the fabrication of hexagonally-ordered bowl-shaped and micro-cavity titanium dioxide (TiO_2) structures for water purification. Generally, close-packed polystyrene(PS) spheres monolayer was prepared by air-water interface self-assembly method. TiO_2 coating was prepared on the as-prepared close-packed PS spheres monolayer by manual painting method. Two types of TiO_2 bowl-shaped film was conducted with TiO_2 sol-gel and TiO_2 nanoparticle solution through annealing. During the transfer of the PS spheres monolayer on the glass slide, coconut oil was used to change the surface tension and enhance the quality of close-packed monolayer. In the latter part of the report, various discussion on the pattern with recommendations will be emphasized so the fabrication process could be easier for its versatility in different research fields, such as water purification of polluted water.

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TABLE 1 Raman shift corresponding to the active modes of TiO₂ anatase phase

Chapter 1 Introduction

1.1 Large contact area between water and TiO₂

TiO₂ is already a very popular material in photocatalyst in a variety of applications and products in the water purification [1]. The higher efficiency of photocatalysis can be achieved by enhancement of the cross-section area between the TiO₂ thin film and the polluted water. In this report, the preparation of close-packed polystyrene(PS) spheres monolayer was conducted through air-water interface self-assembly method. After TiO₂ coating on the close-packed PS spheres monolayer and the removal of PS spheres, a bowl-shaped TiO₂ thin film was fabricated. As the PS spheres are the support and they provide large area.

1.2 Objectives:

The main objectives of this project include:

- (1) Fabrication of close-packed PS spheres monolayer;
- (2) Fabrication of TiO₂ sol gel;
- (3) Fabrication of bowl-shaped TiO₂ monolayer;

1.3 Organization

This report is structured as follows: Chapter 2 gives a brief review of two popular methods in the fabrication of PS spheres monolayer. Chapter 3 presents that the fabrication procedures, including the fabrication of PS spheres monolayer, TiO₂ sol-gel, TiO₂ nanoparticles solution and TiO₂ bowl-shaped film. Chapter 4 discusses the results from the fabrications of Chapter 3. Chapter 5 gives a summary of this project and future recommendation.

Chapter 2 Literature Review

2.1 Spin Coating

Spin coating is a micro process to fabricate a uniformly thick film on the substrate surface when applying a high spin speed [2]. There are some parameters to determine the thickness of coating such as surface tension and viscosity.

The spin-coating method has been used to fabricate PS spheres monolayer on the substrate [3]. First, PS suspension was dipped on the glass substrate. Then the substrate was spun at a certain speed with suitable spin revolution. Finding the suitable spin speed for desired product needs lots of practices and experiences. Therefore, spin coating is more suitable to fabricating liquid material and multilayer on the substrate.

2.2 Air-water interface self-assembly method

The air-water interface self-assembly method is a highly efficient method to fabricate PS spheres monolayer [4]. The strong binding of the particles involves only minimization of the interfacial energy [5]. A free energy difference will be activated because of the change in total interfacial energy when the PS spheres monolayer move

to the air-water interface. As figure 2.1 shown, due to the attraction, the PS spheres will be assembled together by themselves. Surfactant is needed to activate the second self-assembly [6]. Surfactant can change the tension of the surface and the force can push the PS spheres closer and form a close-packed pattern. The quality of PS spheres monolayer depends on many parameters in the air-water interface self-assembly. For instance, the size of PS spheres, the concentration of PS spheres and the concentration of surfactant [7].

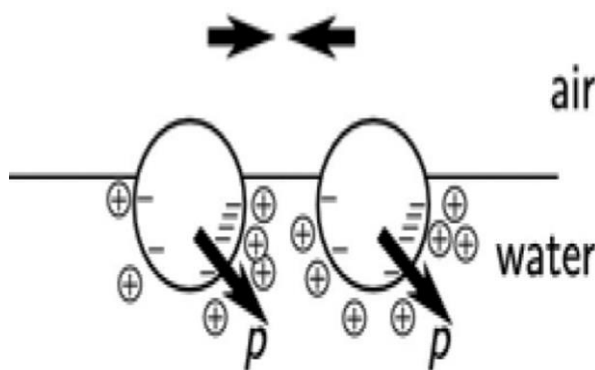


Figure 2.1 The movement of PS spheres on the air-water interface [8].

2.3 Manual painting method

Manual painting method is significantly more precise and faster than the hand painting process [9]. And it has been used for catalyst coating [10] and it provides a high precision in catalyst loading with high reproducibility [11]. As photocatalyst needs

to be coated in this project, manual painting method is a top priority.

2.4 Application of close-packed PS spheres monolayer

Beside in water purification, the usage of close-packed PS spheres monolayer is widely applied. For example, solar cell [12], LED [13], photonic crystals [14], nickel hydroxide electrode [15] and electrochromic [16] etc. In many applications, close-packed PS spheres monolayer is used as a template. It is easy to remove relatively, so the TiO_2 pattern would be made as the complementary site when PS has been removed. Then TiO_2 network pattern would thus provide large contact area which facilitates higher efficiency of many chemical process, such as water purification.

Chapter 3 Fabrication Procedures and Experimental Setups

3.1 Fabrication of PS spheres monolayer

In this project, the most difficult part is the fabrication of PS spheres monolayer.

Spin coating is the first choice as it helps deposit a very uniform pattern.

Manual painting is alternative method as it is easy to conduct. And the air-water interface self-assembly method used in this project will be emphasized due to its ease and low cost.

3.1.1 Material Preparation

The diameter of PS spheres used in this project is $3.3\mu\text{m}$ long and its wt% is 13%.

1 cm^2 glass should be prepared as the substrate of the PS spheres monolayer. Coconut oil solution was made by mixing 1 ml coconut oil and 15 ml DI water. Before the above procedures, the glass slide need to be cleaned in Acetone, IPA(isopropylalcohol) and DI water in 10-min ultrasound bath respectively.

3.1.2 Procedures

1. Drop 0.5 μl PS spheres on the DI water and wait until the PS spheres form a monolayer. A thin and uniform layer of PS film could appear on the DI water.
2. Drop 0.1 μl coconut oil solution on the water and wait until the PS spheres get close.
3. Dip a 1 cm^2 glass into the coconut oil solution.
4. Use the dipped glass to fish up the PS spheres monolayer.
5. Do not move it until it is dry.

3.2 Fabrication of TiO_2 sol-gel

TiO_2 sol-gel is a common material used to fabricate a TiO_2 thin film. And the method used in this project comes from other's paper [17].

3.2.1 Material preparation

As the area of TiO_2 film is 1 cm^2 , a few TiO_2 sol-gel is enough. In this project, 15 ml TiO_2 sol-gel were conducted. A 15 to 20 ml glass bottle was washed by ethanol before use. 1.32ml tetrabutyl titanate with purity 97%, 0.32ml diethanolamine $\{\text{HO}(\text{CH}_2)_2\text{NH}_2\}$, 0.15ml DI water and 13.2ml ethanol with purity 99.7% were need. The quantity of each component depends on how much sol-gel needed, they could be

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determined by the ratio.

3.2.2 Procedures

1. Add the mixture of 10.2 ml ethanol and 0.36ml diethanolamine into the empty glass bottle at 40°C water bath and stir 20 mins with a magnetic stirrer.
2. After 20 mins, add 1.32ml tetrabutyl titanate and stir 50 mins with a magnetic stirrer.
3. After 50mins, add 3ml ethanol and 0.15ml DI water and constant stirring and aging for 24 hrs.

3.3 Fabrication of TiO₂ nanoparticle solution

The method used in this project is based on other's paper [18].

3.3.1 Material preparation

12 g TiO₂ powders with mean size of primary particles approximately 25 nm, 125ml DI water, 0.4 ml acetylacetone, 0.2ml Triton X-100 and 2.4g polyethylene glycol should be prepared.

3.3.2 Procedures

1. Add the mixture of 12g TiO_2 powders and 120 ml DI water into the empty clean glass bottle with magnetic stirring.
2. After the powders were totally dispersed, add 0.2 ml Triton X-100.
3. Add 2.4g polyethylene glycol into the solution and keep stirring over a night.

3.4 Fabrication of TiO_2 thin film

There are many methods to fabricate the TiO_2 thin film, the method used in this project is manual painting and annealing.

3.4.1 Material preparation

The main materials are PS spheres monolayer, TiO_2 nanoparticles solution and TiO_2 sol-gel.

3.4.2 Procedures

1. Drop 1ml TiO_2 sol-gel on the PS spheres monolayer and apply manual painting method to form a coating.
2. After it is dry, put into the oven. Annealing with 500°C in 2 hours.

3. Replace the TiO_2 sol-gel with TiO_2 nanoparticles solution and repeat the procedures above.

Chapter 4 Results and Discussion

4.1 Fabrication of PS spheres monolayer

From figure 4.1, the upper left side is the glass substrate, it shows that there is only one layer on the substrate. The pattern appears relatively close-packed. The defects between the PS spheres are difficult to avoid. From figure 4.2, the size of PS spheres is about $3.3\mu\text{m}$ which is fit to the trade description on the product. The product fabricated through air-water interface self-assembly method could be improved based on experience of each failure.

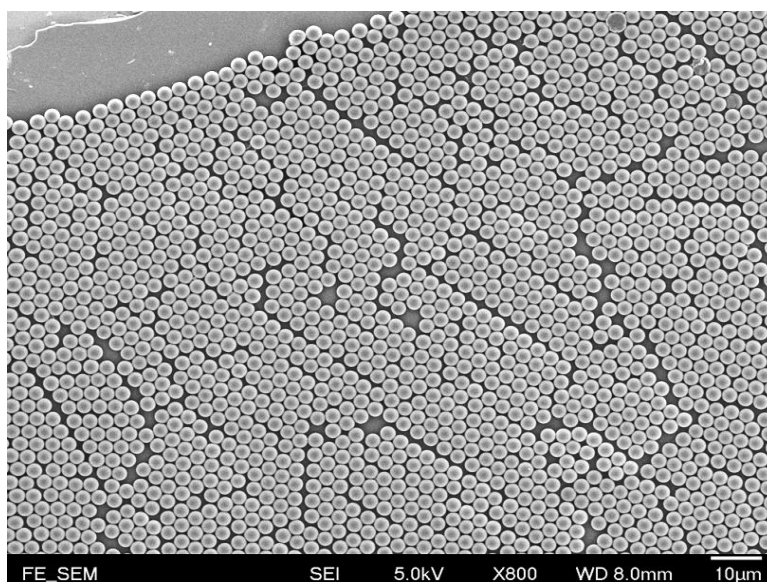


FIGURE 4.1 SEM image of PS spheres monolayer.

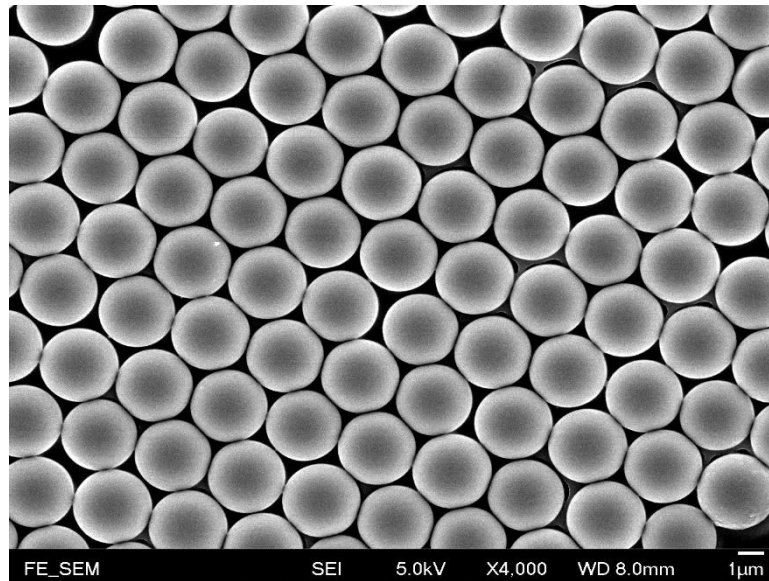


FIGURE 4.2 Magnified SEM image of PS spheres monolayer.

The spin coating method was conducted first and the results were not satisfactory. From figure 4.3 and figure 4.4, the pattern is not close-packed and it is not a monolayer. Even it is a monolayer, the pattern is not as close-packed as the fabricated by air-water interface self-assembly method. In addition, the spin speed which determines the thickness of the coating is difficult to control. To form a monolayer, the spin speed should be optimized to ensure all the PS spheres separating. The spin speed cannot be too fast, and it will not form a close-packed pattern. The thickness of PS spheres monolayer in this project should be $3.3\text{ }\mu\text{m}$, which is same as the diameter of a single isolated PS sphere.

The spin speed, acceleration and the hold time vary with different materials and

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lots of trials need to be demonstrated to find out the suitable parameters. It is time-consuming and expensive compared with air-water interface self-assembly method.

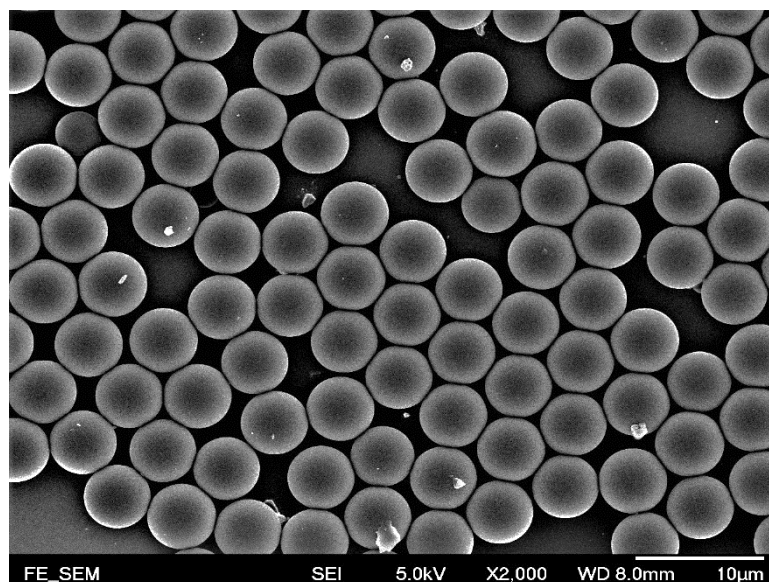


FIGURE 4 SEM image of spin coating fabrication.

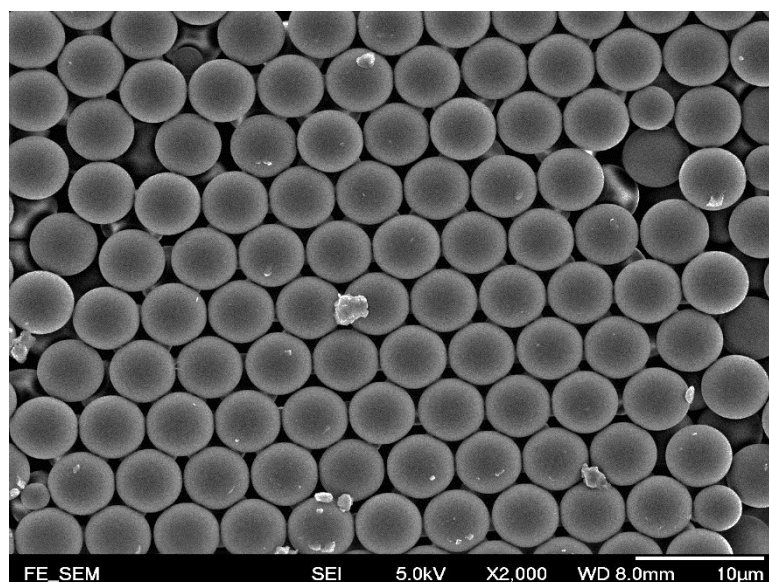


FIGURE 4.4 SEM image of spin coating fabrication.

The second method conducted in this project is air-water interface self-assembly

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method. The special treatment in this method is adding hydrophilicity to the substrate [19]. The glass substrate should be dipped in a piranha solution at 90°C in 1.5 hours to reduce the contact angle between water and glass. The prediction is ideal that the water will flow out and the PS spheres will remain on the glass when the glass gets close to the PS spheres monolayer on the water surface.

The reality in this project is that the pattern was easily broken when the glass was leveled up manually in the water. As figure 4.5 shown, the pattern will separate as the tension of water flow out of the glass is large than the force to maintain the PS spheres.

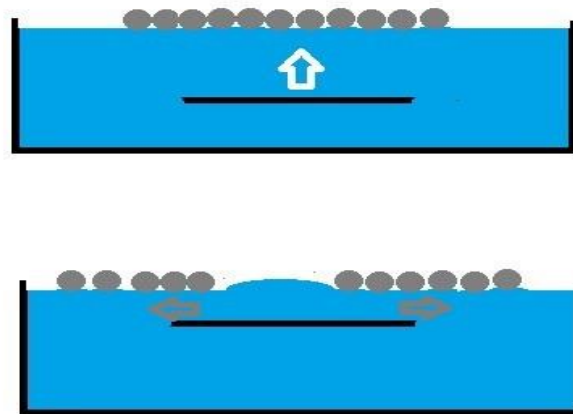


FIGURE 4.5 The situation appears when a hydrophilic glass moves close to the PS monolayer on water face.

4.2 Sputtering

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After the fabrication of close-packed PS monolayer, sputtering was conducted as a trial to form a TiO_2 coating on the PS monolayer. It failed and the TiO_2 target was broken as figure 4.6 shown. It was told that oxide target is not suitable for sputtering. The way to avoid broken is purchasing a piece of metal and sticker to the bottom of the oxide target but the price is doubly higher.



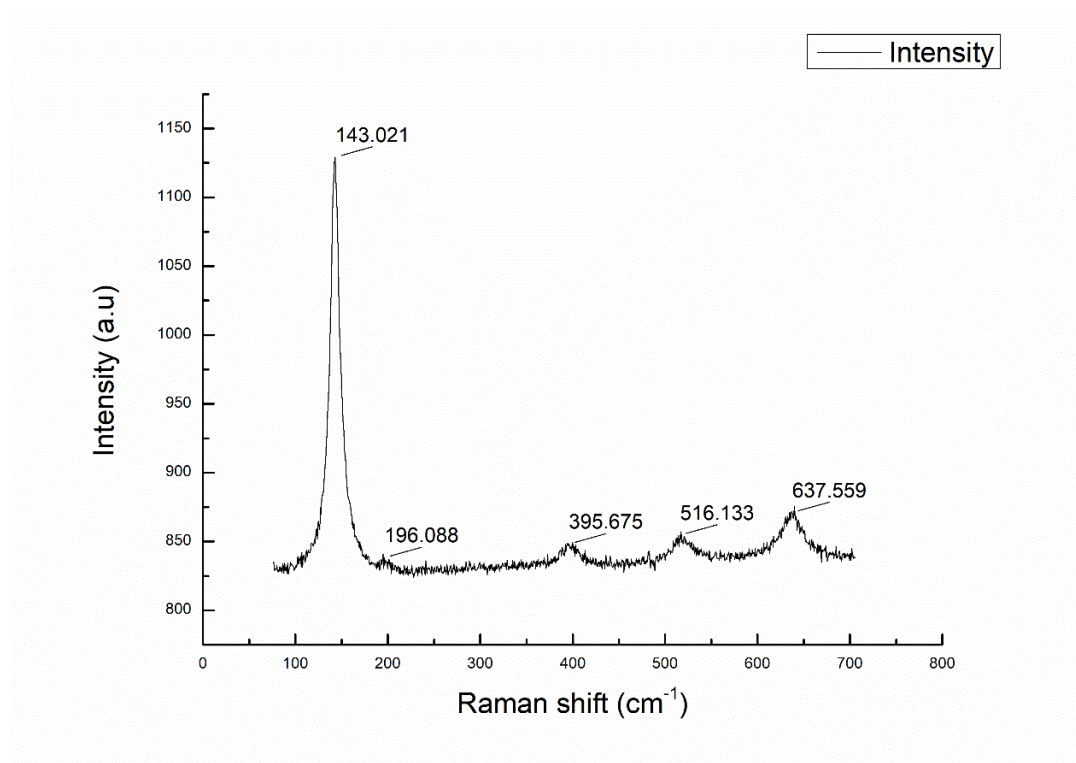
FIGURE 4.6 Broken TiO_2 target.

4.3 The fabrication of TiO_2 sol-gel

The fabrication of TiO_2 sol-gel highly depends on the purity of materials used. To confirm the quality of TiO_2 sol-gel, Raman spectroscopy was conducted with 533nm. TiO_2 in anatase phase has five Raman active modes as shown in Table 1 [20]. And as the figure 4.7 shown, the TiO_2 sol-gel made in this project is the TiO_2 in anatase phase.

TABLE 1 Raman shift corresponding to the active modes of TiO₂ anatase phase

	Raman shift (cm ⁻¹)	Assignment
Anatase	145	Eg(v ₆)
	199	Eg(v ₅)
	396	B ₁ g(v ₄)
	517	B ₁ g(v ₂)/A ₁ g(v ₃)
	638	Eg(v ₁)

**FIGURE 4.7** Raman spectrum of TiO₂ sol-gel.

4.4 The fabrication of TiO₂ bowl-shaped film

The TiO₂ bowl-shaped film was conducted through annealing. The PS spheres were removed in elevated temperature and the TiO₂ bowl-shaped film remained. Figure 4.8 shows the TiO₂ nanoparticles coating was not uniform and it was hard to avoid as it was made through doctor-blade method. Figure 4.9 and 4.10 shown that there are lots of cavity remained. Some of the area were covered by TiO₂ nanoparticles. In figure 4.11 and 4.12, there are cavities inside the covered area. It could be attributed to the non-uniform coating. The gases might go out from the neighbor cavities as the nanoparticles surrounding between PS spheres was thinner than the covered one.

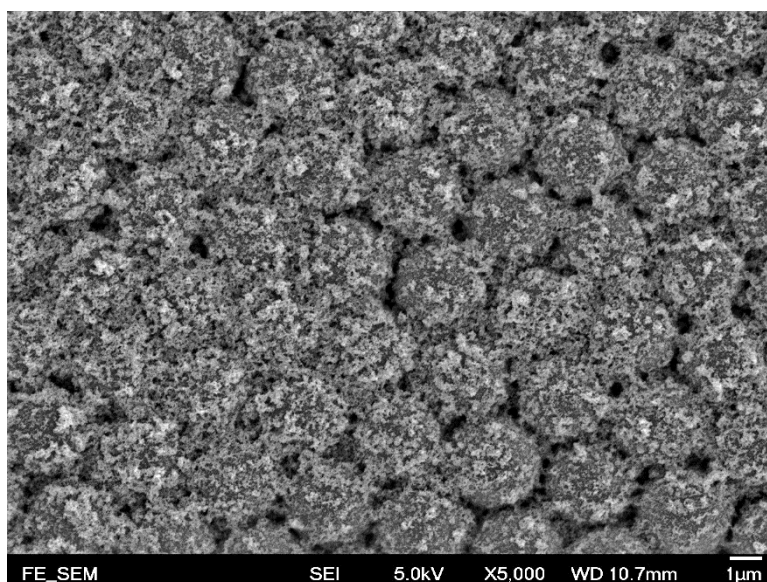


FIGURE 4.8 SEM image of the PS spheres monolayer coating with TiO₂ nanoparticles.

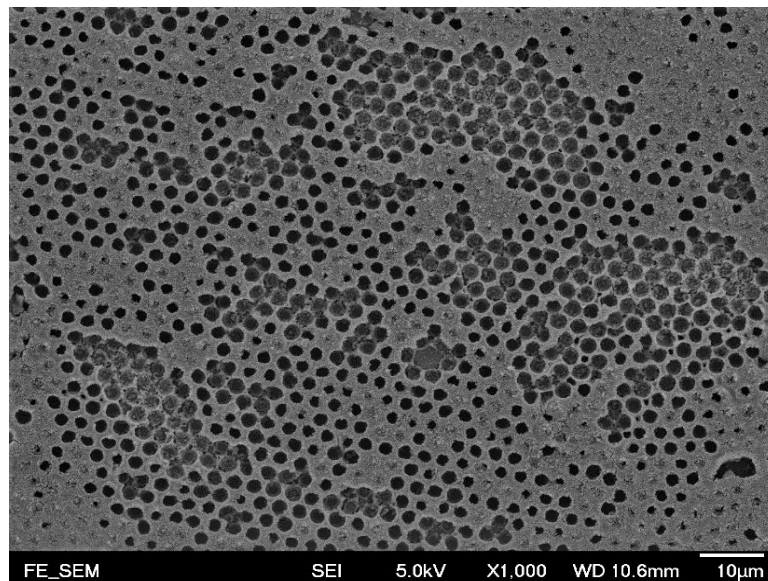


FIGURE 4.9 SEM image of TiO₂ bowl-shaped film made by TiO₂ nanoparticle solution.

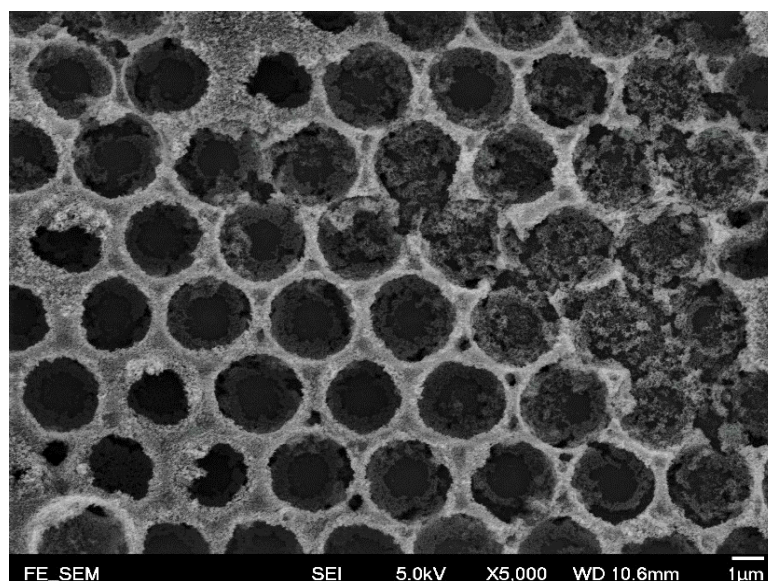


Figure 4.10 Magnified SEM image of TiO₂ bowl-shaped film made by TiO₂ nanoparticle solution.

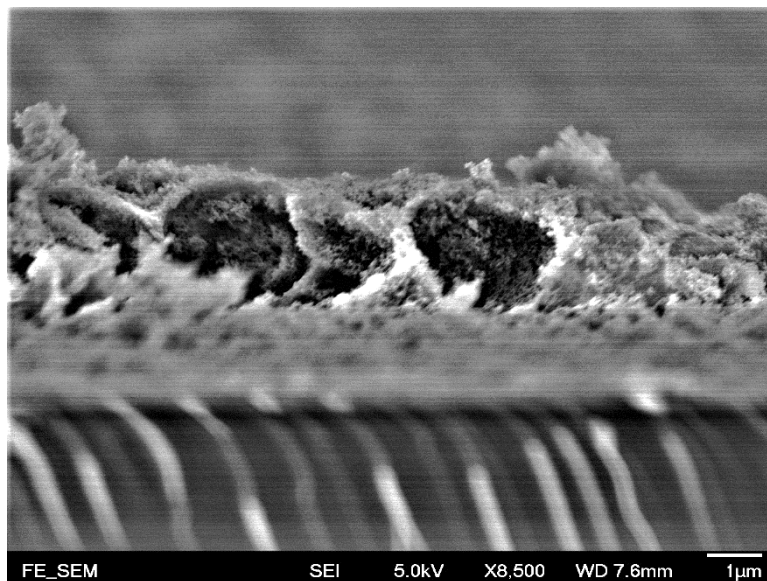


FIGURE 4.11 SEM image of the cross-section area of TiO₂ nanoparticles bowl-shaped film.

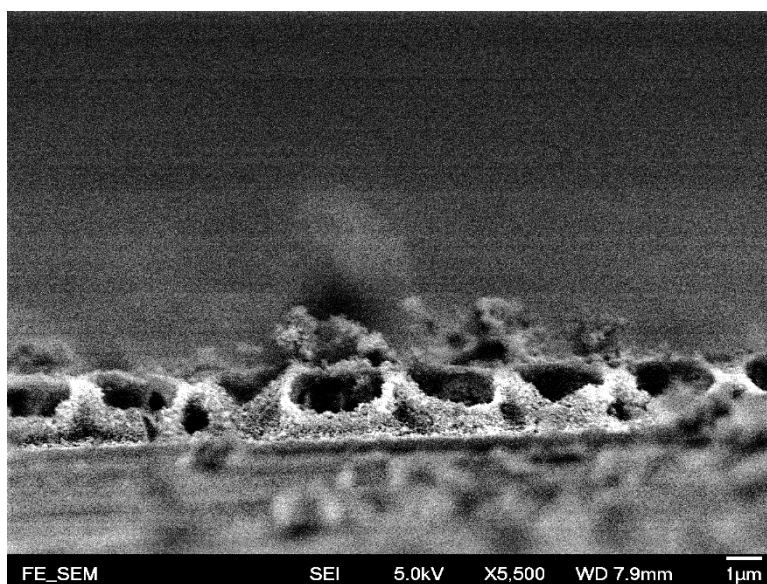


FIGURE 4.12 SEM image of the cross-section area of TiO₂ nanoparticles bowl-shaped film.

In figure 4.13, the pattern of TiO₂ sol-gel bowl-shaped film is as predicted. With

the cross-section area image shown at figure 4.14, there are cavities.

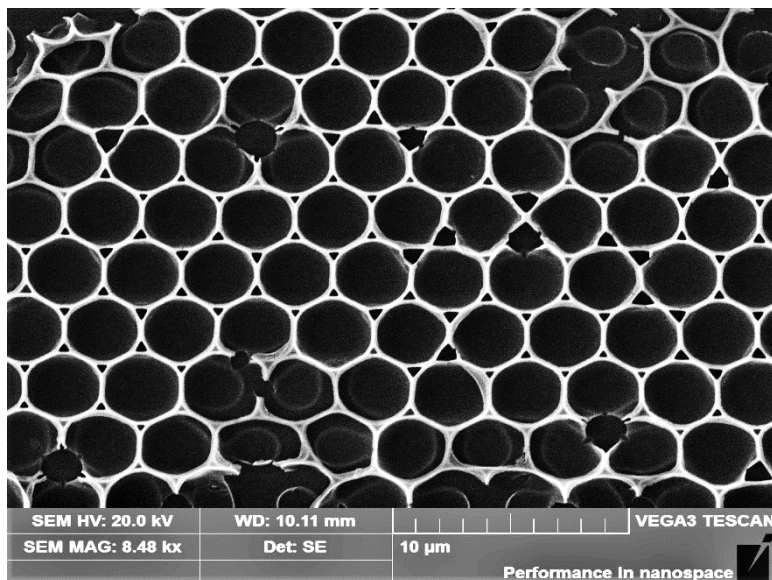


FIGURE 4.13 SEM image of the TiO_2 sol-gel film.

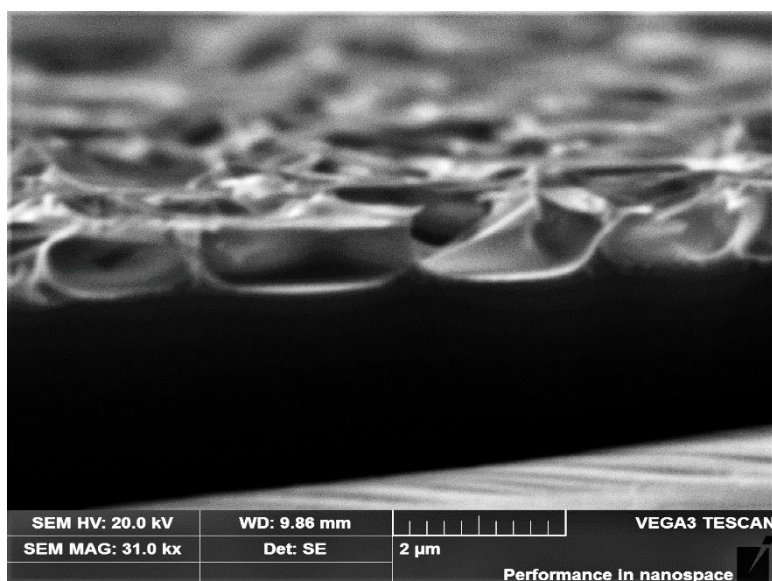


FIGURE 4.14 SEM image of the cross-section area of TiO_2 sol-gel film.

4.5 Defects of PS spheres monolayer.

The main defects of PS spheres monolayer are non-dispersed and multilayer.

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Some of the PS spheres are sticker together before dropping on the water surface which leads to the final non-dispersed and multilayer. The second reason might be the non-steady substrate when it is transferring. Even a small vibration would cause a huge effect to the PS spheres at water surface. And it leads to the multilayer.

4.6 Defects of TiO₂ bowl-shaped film

Both two types of bowl-shaped film have defects after annealing. Compare figure 4.7 and 4.9, there is a uniform pattern before annealing. And the defects are clearly not caused by the defects of PS spheres monolayer. In figure 4.15, there are tidy and uniform marks of PS spheres which reveals there was a monolayer before. The defects might be caused by the dust inside the oven.

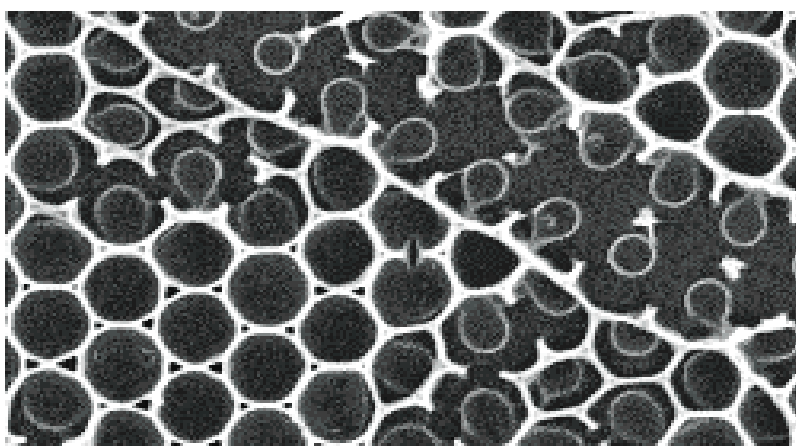


FIGURE 4.15 SEM image of defects on TiO₂ sol-gel bowl-shaped film.

4.7 The usage of Coconut oil

Coconut oil is one of the surfactants and detergent with lipophilicity [21]. It can change the surface tension and cause the second self-assembly of PS spheres on the water. With its lipophilicity, the PS spheres monolayer was shielded and it leads the transfer procedure easier.

4.8 Annealing

Annealing can enhance the photocatalytic performance of TiO_2 [22]. The optical indirect band gap decreases with the increase of the annealing temperature. Annealing is also the method to remove PS spheres and coconut oil between the PS spheres. The typical melting point of PS spheres and the boiling point of coconut oil are 240°C and 171°C [23] The annealing temperature 500°C is much higher than that.

Chapter 5 Conclusion and Future Recommendation

5.1 Conclusions

In the project, close-packed PS spheres monolayer was conducted through air-water interface self-assembly method. TiO_2 nanoparticle solution and TiO_2 sol-gel were coated on the PS spheres monolayer through manual painting method. TiO_2 bowl-shaped films were conducted by annealing. Only cavities remained after annealing. And the cavities can enhance the contact area between the water and the TiO_2 .

5.2 Future recommendation

In the fabrication of PS spheres monolayer part, the preparation of PS spheres should be better. Before dropping the PS spheres on the air-water interface, the PS spheres should be dispersed individually. If they are sticker together, it will be a defect in the next experiment step and leave a large defect in the final product. Long time ultrasound bath is a suggestion. Using the long-time vibration to separate the PS spheres. Another suggestion is spin coating [24]. Spin coating can ensure all the PS spheres are

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separated at high spin speed. However, it cost more PS spheres. A small machine arm could be applied in the transferring part. As mentioned that a very small vibration could affect the close-packed pattern and cause defects. Machine arm can ensure the substrate move to the PS spheres monolayer at a constant speed and avoid vibrations.

In the fabrication of TiO_2 sol-gel part, the purity of materials is important. To best way is to use the new chemical. During the procedures in fabrication, adding solution should be as fast as possible to ensure the components less contact to the air. The water in air would affect the purity of materials.

In the fabrication of TiO_2 bowl-shaped part, the remove of PS spheres is easy. However, more consideration should be taken to prevent the defects from the layer. Annealing is an easy method. And it is believed that there is a better result in a clean oven. Another suggestion is to immerse in chloroform [25] because PS spheres and coconut oil would be dissolve in chloroform.

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