Synthesis Of Nano-Porous Strontium Titanate Materials for Photocatalysis

Yuzhe Pei *

Faculty of Mathematical and Physical Sciences, University College London, London, United Kingdom

* Corresponding Author Email: peiyzhe@outlook.com

Abstract. Strontium titanate semiconductor materials have been widely used in the field of photocatalysis due to their high dielectric constant and band gap. Uniform, micron-sized $SrTiO_3$ particles didn't tend to aggregate, and the low surface area of larger particles could be improved by incorporating porous structures, thus offering superior performance for a range of applications. In this study, six mainly methods to prepare nano-porous $SrTiO_3$ particles were showed and discussed. The methods include hydrothermal method，sol gel method, chemical co-precipitation method, high temperature solid phase method, hard template method and soft template method. These processes and their advantages and disadvantages were analysised.

Keywords: Strontium titanate; hydrothermal method; nano-porous structure; template method; photocatalysis.

1. Introduction

The rapid development of technology has led to a great leap in human society, but urban sewage and factory discharge of wastewater have also led to a series of serious negative problems [1] such as increasingly serious environmental pollution, energy and resource shortages, which have become a serious challenge facing the world today. The photocatalytic degradation of organic pollutants [2] should be of great significance and role in solving these problems. Photocatalytic materials, such as perovskite strontium titanate [3], can convert solar energy into electrical or chemical energy, which is the key to the efficient utilization of solar energy. Therefore, the development of efficient and practical photocatalyst is urgent and has extremely important academic and application value.

Strontium titanate ($SrTiO_3$) is a typical perovskite structured material. On March 9, 2023, the team led by The Ruihua from West Lake University published their research findings in Nature [4], discovering for the first time the appearance of a photocathode on the surface of strontium titanate crystals, which generates electron clouds on the material surface through light excitation. The research results indicate that strontium titanate is the only material in semiconductor materials where photoelectrons can jump out of the bulk and into space. The discovery of this phenomenon also provides more fundamental theoretical support for the application of strontium titanate in the field of optoelectronics. Due to its excellent optoelectronic properties, strontium titanate is widely used in energy [5], environment [6], electronics, machinery, medical, and ceramic industries [7]; Especially in the fields of photocatalytic water splitting for hydrogen production [8], photovoltaic power generation, and photocatalytic degradation of organic pollutants [9], it has received widespread attention.

Porous structures can enhance loading capacity and facilitate microscale transport of ions and electrons. Nano porous strontium titanate can significantly enhance the photocatalytic activity of $SrTiO_3$[10]. In recent years, many new methods for preparing nano porous $SrTiO_3$ have been studied both domestically and internationally [11]. In addition, doping modification can expand the light absorption range of $SrTiO_3$ to the visible light region [12]. The large pore array is conducive to efficient material transfer at the interface, while highly active catalytic points located on the mesoporous wall can be used for material adsorption and reaction. Therefore, exploring the preparation of nano porous $SrTiO_3$ and its doping modification has important theoretical significance and practical application value.

2. Hydrothermal method

The hydrothermal method for preparing strontium titanate is achieved by fully dissolving different ratios of raw materials, placing the mixture into a high-pressure reactor, and then preparing it at a certain temperature and pressure. Due to its advantages of low reaction temperature, simple operation, and easy process control, hydrothermal method is currently a common method for synthesizing nanoscale porous $SrTiO_3$[11]. Researchers [13] added titanium dioxide powder to a strontium source and controlled the size of strontium titanate grains by changing hydrothermal time, temperature, pH value, and reactant concentration, thereby adjusting the pore size. The main formation process was that when TiO_2 was added to Sr $(OH)_2$ solution, the surface of TiO_2 will adsorb OH^- and H_2O molecules in the solution through covalent bonding. OH- and H2O react with Ti ions in TiO_2 or O atoms in the lattice to form $HTiO^{3-}$, which then reacted with Sr^{2+}ions to form $SrTiO_3$.

In Wei [14] et al.'s study, adding polyvinyl alcohol to a hydrothermal reaction solution resulted in mesoporous $SrTiO_3$ microspheres with a specific surface area of 264.9 m^2/g and uniform dispersion.

Xu Cunying et al. [15] used $TiCl_4$ and Sr $(NO_3)_2$ as the basic raw materials, KOH as the mineralizer, and dodecylbenzenesulfonic acid (DBS) as the surfactant to obtain SrTiO3 nanoparticles with an average particle size of 120nm coated with DBS by hydrothermal method. The average thickness of the coating film was 6nm, and the particle shape was regular with good monodispersity.

Another researcher, Kuang et al. [16], analyzed the morphology and crystal planes of $SrTiO_3$ prepared under different hydrothermal conditions and time conditions, and found that this formation process was closely related to topological orientation transformation and oriented Ostwald ripening mechanism. Through photocatalytic experiments, researchers found that the hydrogen production efficiency of porous strontium titanate prepared under hydrothermal conditions of 150℃ was the highest.

Dong et al. [17] successfully prepared porous $SrTiO_3$ microspheres by hydrothermal method, which can achieve 100% degradation of Rhodamine B under UV light irradiation for 20 minutes.

3. Sol gel method

In the study by Zhang Wenkui et al. [18], strontium nitrate was first dissolved in a solution of glacial acetic acid. Then, tetrabutyl titanate was added to anhydrous ethanol and thoroughly mixed. The two solutions were mixed dropwise and stirred continuously. After mixing, add glycerol and stir for 0.5 hours. Then, let it stand in a 60℃-water bath and wait for gelation. Finally, fully calcine in a muffle furnace to produce $SrTiO_3$ powder. Through specific experiments, it has been shown that pH value plays an important role in the formation of colloids, and no colloids will be produced when $pH \leq 1$ or $pH \geq 6$; On the contrary, the gelation time is inversely proportional to the pH value.

Zhou Zukang et al. [19] believed that hydrolysis and polycondensation of metal alkoxides were extremely important reasons for the final formation of sol, and properly controlling the temperature below 40℃ when the sol was converted to gel could significantly improve the speed of gel formation. The $SrTiO_3$ powder formed by this method has a uniform particle size distribution, and its crystal shape, particle size, and lattice constant are directly proportional to the temperature during heat treatment.

In sol gel synthesis, the porous structure can be controlled by the concentration of template. Finally, methods such as calcination can be used to remove the template agent and retain its shape in the material, forming a porous structure [20]. Some researchers [21] added different amounts of template PEG-4000 in the sol gel method to carry out porous modification, which can not only increase the specific surface area, but also change the phase of strontium titanate from Sr_2TiO_4 to $SrTiO_3$. The final research results indicate that when 1.5g PEG-4000 is added [22], the particle size of strontium titanate porous material is significantly reduced, and the specific surface area and crystallinity are significantly increased.

4. Chemical co-precipitation method

The chemical co precipitation method is a method of dissolving soluble metal ions in a solvent according to a stoichiometric ratio, and then adding a precipitant that can form co precipitation with the metal ion, so that the metal ion can precipitate in the form of hydroxides, carbonates, or insoluble hydrates, and ultimately obtain the desired product. In the study by Zhu Qi'an et al.[23], a certain volume of $SrCl_2$ solution and $TiCl_4$ solution were taken and placed in a separating funnel at a molar ratio of 1.02:1 between $SrCl_2·6H_2O$ and $TiCl_4$; Take a certain volume of $(NH_4)_2CO_3$ solution with a molar ratio of 1.40:1 and $SrCl_2·6H_2O$ into a three necked flask; After adding a certain volume of ammonia, turn on the stirrer and stir. Add the mixed solution of $TiCl_4$ and $SrCl_2$ from the separating funnel dropwise into a three necked flask to generate a co precipitation of $SrCO_3$ and $TiO(OH)_2$. Among them, in order to precipitate $Sr^{2+}$and TiO^{2+} simultaneously and completely, $NH_3·H_2O$ was used to adjust the pH value above 10.5 during the reaction process. After reacting at room temperature for 4 hours, the co-precipitate was filtered and washed until Cl^- could not be detected with $AgNO_3$ solution. Then, it was dried, ground and placed in a muffle furnace for 4 hours at 900°C to obtain $SrTiO_3$ powder.

The research of Fang Huihui et al. [24] shows that the precipitation reaction temperature and time have a certain impact on the product concentration: excessive temperature can cause the precipitation agent to decompose, and Sr^{2+} cannot precipitate into $SrCO_3$, resulting in a decrease in product purity. The best reaction temperature should be controlled at around 25℃; A short reaction time can lead to incomplete precipitation of Sr^{2+}, affecting purity, while the opposite has no significant effect. After multiple experiments, it has been proven that controlling the reaction time at 1.5 hours is appropriate. The calcination temperature and time also have an impact on purity: too low temperature and too short time can lead to incomplete reaction, which may result in the formation of strontium titanate; If the temperature is too high and the time is too long, there will be agglomeration sintering phenomenon; The optimal roasting temperature is controlled at 920 ± 20℃ and time is 2.5 hours.

Research by Lu Hongxia et al. [25] has shown that compared with high-temperature solid-phase method, chemical co precipitation method requires lower calcination temperature and shorter calcination time. They studied the use of Na_2CO_3 and NaOH as precipitants to prepare nano $SrTiO_3$ particles with small particle size, high purity, and good crystal structure. This method can accurately control the particle size distribution uniformity of the product and has a good guiding effect on the preparation of high-purity $SrTiO_3$ nanomaterials.

Zhang Hao et al. [26] found through experiments that strontium titanate powder with a particle size of about 60 nm was synthesized using chemical co precipitation method using metatitanic acid, hydrogen peroxide, ammonia, and strontium nitrate as raw materials. The metatitanic acid was washed with 0.5% ammonia solution to remove most impurities and ensure the purity of the product.

5. High temperature solid-phase method

High temperature solid-phase method is a method of high-strength mechanical grinding several oxides according to a certain stoichiometric number, and then forging the ground mixture at high temperature (800°C~1500°C) to obtain the desired product. Yang Shuibin et al. [27] chose to uniformly mix SrO(or $SrCO_3$) and TiO_2 powders, then press them into sheets and calcine them at high temperatures above 1000°C for several tens of hours to prepare strontium titanate powder.

The entire preparation process can be divided into two parts: (1) the formation of $SrTiO_3$crystal nuclei, followed by the formation of $SrTiO_3$ layers; The difficulty in this layer lies in the significant structural differences between SrO, TiO_2, and $SrTiO_3$. In order to further generate $SrTiO_3$, a large number of chemical bond breaks and recombinations will occur, and atoms also need to migrate. (2) TiO^{4+} and Sr^{2+} diffuse into the crystal phases of SrO and TiO respectively (passing through the crystal phases of $SrTiO_3$) [28].

Both of these parts require high energy, so they need to be carried out at high temperatures. However, the high-temperature solid-phase method has problems such as difficulty in mixing uniformly and incomplete reaction.

Liu Guya [29] successfully prepared cubic crystalline $SrTiO_3$ using high temperature solid-phase method, with a particle size of about 125nm and high purity, without impurities such as TiO_2 and $SrCO_3$. The advantage of high-temperature solid-phase method is that it does not require the use of solvents, thereby reducing the pollution of solvent volatilization on the environment and promoting the large-scale production of $SrTiO_3$.

In the research of Liu H [30], $SrTiO_3$ was synthesized by mixed hydroxides (50 g NaOH/KOH 51.5:48.5, analytical grade) in a 50 mL Teflon vessel. Then the vessel was preheated for 3 hours under 205°C until the hydroxides were completely molten. Then 3 mmol $SrCl_2$ (analytical grade) and 3 mmol TiO_2 (Degussa P25, Frankfurt, Germany/nanoparticle/nanosheet/TiO^{2-}μm) were added to the molten hydroxides. A Teflon bar stirred the solution to homogenize the mixture, then heat the vessel at 205°C for 12 hours. After the vessel was cooled down to room temperature, the hydroxides were removed carefully by distilled water at different temperatures (0°C, room temperature: 20-25, 60, and 80°C).

6. Hard template method

Kiyofumi Katagiri et al. [31] successfully synthesized mesoporous silica composite strontium titanate nanocube crystals using mesoporous silica as a template and oleic acid as a surfactant by hydrothermal method. The analysis results show that strontium titanate crystals grow uniformly in mesoporous silica. BET analysis shows that the specific surface area of the composite material is $543m^2/g$, slightly lower than the original mesoporous silica material's $835m^2/g$. The prepared nanocomposite porous material exhibits significantly higher photocatalytic activity and decomposition rate than traditional strontium titanate materials, mainly due to the high specific surface area provided by the mesoporous silica matrix.

Wenjun Dong [32] and other experimenters successfully synthesized a porous strontium titanate material by hydrothermal method using the template of titanate nanotubes and taking strontium chloride as strontium source. By flexibly adjusting reaction parameters and other conditions, the research team easily mastered the morphology and pore size of strontium titanate spheres. The experimental results indicate that the specific surface areas of porous and solid strontium titanate spheres prepared using titanate nanowire precursors are 25.8 and $12.1m^2/g$, respectively. These porous strontium titanate spheres exhibit excellent adsorption and photocatalytic properties.

Kuang et al. [33] synthesized porous $SrTiO_3$ using a template method. Layered protonated titanate graded spheres (LTHSs) were used as templates to prepare a three-dimensional porous strontium titanate structure formed by self-assembly of $SrTiO_3$ single crystals using a hydrothermal method. The prepared $SrTiO_3$ single crystals all have a homogeneous cubic phase structure, with a size of approximately 60-80 nm and exposed crystal faces of (100) planes.

Some researchers [10] used agarose gel containing strontium carbonate crystals as hard template to successfully prepare three-dimensional porous strontium titanate microspheres with a diameter of about 17μm. The microspheres have mesoporous and macroporous distributions inside, with a mesoporous diameter of approximately 3 nm and a macroporous diameter of approximately 70-150 nm. The analysis results indicate that strontium titanate microspheres are self-assembled from cubic single crystals of strontium titanate, and the highly crosslinked porous microsphere material significantly enhances its light absorption and material adsorption abilities.

Zhengbo Jiao et al. [34] first synthesized titanium dioxide nanotube arrays, and then used hydrothermal method to prepare strontium hydroxide as a strontium source to prepare titanium dioxide nanotube arrays wrapped in strontium titanate thin films and strontium titanate nanospheres, respectively. The research results indicate that the prepared composite material can stimulate the separation of strontium titanate hole electron pairs, significantly enhancing the photocatalytic performance of the material.

Jia Hong Pan [35] and others combined the sol gel method and hydrothermal process. First, amorphous titanium dioxide spheres were synthesized by the sol gel method as precursors and templates. Then, a variety of porous perovskite titanate spheres were prepared by hydrothermal reaction in the alkaline system. And by adjusting the size and porosity of amorphous titanium dioxide sphere precursors, $ATiO_3$ (A=Sr, Ba and Ca) spheres with different sizes and internal structures were successfully synthesized. This porous material has a large specific surface area and a good single crystal nanocube composition.

Zou Xiaoyan [36] used titanate nanotubes as hard templates to prepare strontium titanate nanotube materials under simple hydrothermal conditions, and then prepared porous strontium titanate microsphere materials through a combination of hydrothermal method and calcination. The analysis results show that both strontium titanate nanotube materials and porous strontium titanate microspheres have a large specific surface area, which can provide more active sites, and both have good photocatalytic performance.

7. Soft template method

The specific surface product of porous strontium titanate materials prepared by soft template method is slightly lower than that of porous strontium titanate materials prepared by hard template method. For the preparation of porous strontium titanate, the sol gel method, which is easy to control the pore distribution and size, is also the most popular method.

The preparation method invented by Chen Long et al. [37] used strontium chloride hexahydrate as the strontium source and butyl titanate as the titanium source. $SrTiO_3$ material was prepared by mixing strontium chloride aqueous solution with butyl titanate ethanol solution, separating, washing, drying the product, and calcining it. By adding organic small molecule triethylamine as a soft template in the reaction system to regulate the morphology and structure of $SrTiO_3$, with higher purity and more uniform particle size distribution were prepared.

He Yawen et al. [38] synthesized mesoporous and microporous strontium titanate using carboxymethyl cellulose (CMC) and hexadecyltrimethylammonium bromide (CTAB) as soft templates through hydrothermal method respectively. The average pore size of mesoporous strontium titanate was 10nm, and the average pore size of microporous strontium titanate was 1.89nm. This study indicates that both CMC and CTAB can control the particle size of strontium titanate, and the analysis results also indicate that the synthesized mesoporous strontium titanate material has stronger organic degradation ability.

Sun Xin [39] used strontium nitrate and tetrabutyl titanate as strontium source and titanium source, and prepared strontium titanate precursor by sol gel method. Then, strontium titanate was porous modified by adding PEG-4000, and metal doping was also studied. The research results indicate that PEG-4000 can lead to a decrease in the particle size of strontium titanate porous materials, while the specific surface area and crystallinity are significantly increased. Moreover, at a dosage of 1.5 g, the product exhibits the best photocatalytic performance.

Li Shengfei[12] used butyl phthalate and strontium acetate as strontium source titanium source, and prepared strontium titanate porous film material with multistage pore structure using the sol gel dip pulling method and PEG-2000 template. Research has shown that the porous strontium titanate material prepared with a template agent at 1.2g/100ml exhibits the best photocatalytic performance and a specific surface area of up to 121.9 m2/g.

8. Conclusion

This paper introduced six mainly methods of nano porous strontium titanate, which were hydrothermal method, sol gel method, chemical coprecipitation method, high temperature solid phase method, hard template method and soft template method. Hydrothermal method, sol gel method and chemical coprecipitation method were characterized by mild reaction conditions, low energy consumption and easy process control, among which sol gel method and chemical coprecipitation method produce relatively poor product morphology. The high-temperature solid-phase method had high reaction efficiency, but there were problems such as high energy consumption, high carbon emissions, and irregular morphology. Compared to the soft template method, hard template agents can prepare nano porous strontium titanate products with a more stable structure and more regular pore morphology, but they had the problem of high cost.

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