Synthesis And Environmental Application of Nano Strontium Titanate

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Abstract. In recent years, perovskite compounds have become a hot spot in the field of photocatalysis due to their unique structures. Strontium titanate ($SrTiO_3$) is a typical perovskite composite oxide, which has excellent properties such as high dielectric constant, low loss and good thermal stability, and its applicability in many fields is significantly better than that of traditional materials, and its application prospects are very broad. In this paper, the research progress of the synthesis of $SrTiO_3$ nanoparticles at home and abroad is reviewed. The main methods include hydrothermal method, solid phase method, sol-gel method, chemical co-precipitation method, etc. Among them, the hydrothermal method and the solid-phase method have good industrial application prospect, and the synthesized products are of high quality, low cost, and easy to control the process. Moreover, $SrTiO_3$ has a good application prospect in environmental protection, especially in wastewater treatment.

Keywords: strontium titanate, nanoparticles, synthesis, environmental applications.

1. Introduction

With the rapid development of modern industry, environmental pollution and energy shortage have become urgent problems to be solved today. The photocatalytic technology launched based on solar energy, which has great potential, has become one of the ways to solve the environmental and energy problems that human beings rely on. Converting solar energy into clean energy requires photocatalysts with good performance, so the research on photocatalysts is urgent. $SrTiO_3$ Photocatalyst is a new type of catalyst applied to industry after TiO_2, which is of great significance in environmental protection and new energy development and application.

$SrTiO_3$ has a typical perovskite structure, high dielectric constant, low loss, good thermal stability and other excellent properties, and its applicability in many fields is significantly better than that of traditional materials, and the application prospect is very broad. For example, Xu Jinbo et al. studied $SrTiO_3$ photoelectrochemical cathodic protection materials [1]. Liu Hongbo et al. conducted research on the preparation and dielectric properties of $SrTiO_3$-bismuth electronic ceramics [2]. Wang Di et al. conducted research on the biomimetic synthesis of nano-$SrTiO_3$ and the adsorption application of heavy metals in sewage [3].

$SrTiO_3$ the synthesis method has hydrothermal method [4], solid-phase method, sol-gel method [5], chemical co-precipitation method, etc. The hydrothermal method can directly prepare nano $SrTiO_3$ powder from the liquid phase, with low synthesis temperature, low production cost and high product quality, but the reaction time is long, and the equipment requirements are relatively high. The high-temperature solid-state reaction method has a simple process, short reaction time and easy to control operation, but the product is easy to agglomerate, the particle size is uneven, and the process energy consumption is high. The low-temperature solid-phase method has short reaction time, high yield, low energy consumption, simple process, no solvent for reaction, and less environmental pollution, which is suitable for large-scale production. The sol-gel method has the characteristics of low reaction temperature, simple equipment, small and uniform particle size, and high purity, but there is a certain agglomeration phenomenon between particles and poor sintering. In the process of synthesizing powder by chemical co-precipitation, more substances are added, and impurities are easy to introduce, and the quality of $SrTiO_3$ powder is reduced. In the process of reaction, it is easy to produce agglomeration, and it is difficult to control the particle size.

2. Synthesis method

2.1. Hydrothermal method

Jing et al. used a two-step soft chemical hydrothermal method to synthesize $SrTiO_3$ [6] . The first hydrothermal process uses LiOH, TiO_2, and KOH as reaction raw materials. Plate-shaped ones are obtained under hydrothermal reaction conditions$K_{0.8}Ti_{1.73}Li_{0.27}O_4$ (KTLO). After two acid exchanges in HNO_3 solution, a solid titanate HTTO in tabular form was obtained. In the second step of the hydrothermal process, the intermediate product HTO reacts with strontium hydroxide, the effects of reaction temperature and time on the product are discussed. The results showed that the $SrTiO_3$ powder prepared by soft chemical synthesis method had high purity, small particle size and uniform particle size distribution, and the tabular $SrTiO_3$ powder was successfully synthesized, which effectively improved the product quality.

Xu synthesized $SrTiO_3$ nano powders with different morphologies by hydrothermal method[7] . By hydrothermal synthesis, the product form can be controlled by changing the amount of ethylene glycol (EG). The smaller the amount of EG, the higher the degree of crystallization and the better the photocatalytic performance. Conversely, the higher the proportion of EG in the system, the lower the degree of crystallization. Two hierarchical structures of $SrTiO_3$ with controllable morphology were synthesized by hydrothermal method including echinoid and lychee-like $SrTiO_3$ nanoparticles. After calcination at 800℃, the overall structure of the product has not changed, and the particle size distribution is more uniform, with its particle size distribution at 30~100nm. By adjusting the ratio of the mixed solvents. Compared with sea urchin, lychee-like $SrTiO_3$ powder exhibited higher photocatalytic activity. In addition, by comparing the photocatalytic performance of the samples before and after calcination, it was found that the crystallinity of $SrTiO_3$ powder was higher after calcination, and its photocatalytic performance was also greatly improved.

Zhang Na studied the synthesis of nano-$SrTiO_3$ by oleic acid-assisted one-step hydrothermal method[8]. The effects of Sr/Ti molar ratio, oleic acid concentration and reaction time on the morphology, purity and grain size of nanoparticles were investigated using $Sr\ (OH)_2 \bullet 8H_2O$ as the strontium source and anatase TiO_2 as the titanium source. Among them, the most important influencing factor is the Sr/Ti molar ratio: with the increase of the Sr/Ti molar ratio, the grain size of the product shows an increasing trend; When the Sr/Ti molar ratio is 0.75, the product has high purity and good crystallinity. The optimal process conditions were as follows: molar ratio Sr/Ti=0.75, oleic acid concentration 3%, and reaction time of 30 h. The introduction of oleic acid can regulate the preparation of $SrTiO_3$ nanomaterials with uniform size and regular morphology, and the particle size of the products decreases with the increase of oleic acid concentration, reducing the agglomeration of $SrTiO_3$ nanoparticles.

$SrTiO_3$ with different exposed areas of (100) and (110) facets have been successfully prepared by hydrothermal synthesis [9]. Although the light absorption area and band structure show little difference in the two samples, as proved by the UV-vis absorption spectra and XPS valence spectra, the photoinduced utile nation has been significantly improved by the (110) facet with a larger area. Moreover, combining transient spectroscopy and XPS technology, we provided a clear picture of the difference between the photoinduced carriers properties′ among STO (110)-S and STO (110)-L, which includes the electronic excited states and Eb. As far as we know, the lower binding energy of STEs caused by the (110) facet has not been reported yet. Furthermore, from the probe reaction, STO (110)-L exhibited notably better activity than STO (110)-S toward the photooxidation of Co^{2+} to Co^{3+}, proving the stronger synergistic effect of the (110) and (100) facets, which could provide great potential for integration into the practical green recovery of heavy metal devices. Finally, our work not only enriches the pool of photocatalytic materials based on $SrTiO_3$ but also contributes new insights to metal vacancy and STEs characteristics in $SrTiO_3$.

The preparation of nano-$SrTiO_3$ by hydrothermal method can make the reactants fully mixed and completely react in the liquid phase medium. The hydrothermal method can directly prepare nano $SrTiO_3$ powder from the liquid phase, with low synthesis temperature, low production cost and high product quality, but the reaction time is long, and the equipment requirements are relatively high. The prepared nano-$SrTiO_3$ has high purity, uniform particle size, controllable morphology and crystal plane.

2.2. Solid-phase method

According to Liu Guya [10] 's research, $SrTiO_3$ was prepared by a simple high-temperature solid-state synthesis method for the synthesis and characterization of $SrTiO_3$, and the structure, composition, shape, size and properties of the product were analyzed by XRD, TEM, FTIR and other analytical techniques. The $SrTiO_3$ obtained by reacting at 600 °C for 10 h had good crystallinity. and in its infrared spectrogram. The infrared spectra of other substances were not present, which indicated that the product only contained $SrTiO_3$ and did not contain possible impurities such as TiO_2 and $SrCO_3$, which was also consistent with the characterization conclusion of XRD.

Ding Shiwen[11] $SrTiO_3$ nano powder was prepared by low-temperature solid-state reaction using $TiCl_4$ and $Sr(OH)_2$•$8H_2O$ as raw materials in the preparation of $SrTiO_3$ powder by low-temperature solid-state reaction. XRD and TEM were used to characterize the powders, and the effects of grinding time, reaction time and reaction temperature on the phase structure of $SrTiO_3$ nano powders were studied. Cubic $SrTiO_3$ powder was prepared by low-temperature solid-state reaction, which opened a new synthesis process. This method has the characteristics of high yield and simple process. The results showed that the optimal grinding time, reaction temperature and reaction time for the preparation of $SrTiO_3$ nano powder were 1 h, 40°C and 5 h, respectively. The $SrTiO_3$ nano powder prepared under these conditions is a pure cubic phase $SrTiO_3$ crystal, with uniform square particles and good dispersion, with an average particle size of about 40 nm.

In the paper "Crystal Facet Engineering on $SrTiO_3$ Enhances Photocatalytic Overall Water Splitting," [12] Yang Zhang mentioned that we have successfully fabricated uniform $SrTiO_3$ single crystals exposing tailored {111} facets via a solid-state recrystallization reaction. A novel crystal facet engineering strategy through solid-state synthesis was developed: a low crystalline $SrTiO_3$ precursor was used to improve the solid-state transformation reaction kinetics, and the additive of inorganic Al^{3+} ions was adopted to engineer the crystal surface orientation. Accordingly, the cuboctahedra SrTiO3 single crystals enable the effective anisotropic separation of photoexcited electron‑hole pairs between {100} and {111} facets. The photocatalytic OWS performance increases with the exposure percentage of {111} facets. Owing to the high crystallinity, low defect density, and anisotropic surface structures, the hydrogen evolution rate for the OWS of Cuba-STO reaches 1.55 $mmol \cdot h^{-1}$, 3 times higher than isotropic SrTiO3 enclosed with {100} facets.

The high-temperature solid-phase reaction method is the traditional method for the preparation of $SrTiO_3$ powder. Generally, the powder of SrO (or $SrCO_3$) and TiO_2 is mechanically ground and mixed evenly according to the stoichiometric number, and then calcined for a few hours to dozens of hours at high temperature (above 1000 °C). The high-temperature solid-state reaction method has a simple process, short reaction time and easy to control operation, but the product is easy to agglomerate, the particle size is uneven, and the process energy consumption is high.

The low-temperature solid-state reaction method has the characteristics of short reaction time, high yield, low energy consumption and simple process, because it does not need to add solvents in the synthesis process, it not only effectively avoids the hard agglomeration of powder and the introduction of impurities in the liquid-phase reaction method, but also has little environmental pollution, and does not use solvents in the low-temperature solid-state reaction, which is suitable for large-scale production, and also provides technical support for the industrial application of $SrTiO_3$.

2.3. Sol-gel method

Zhang Wenkui [13] synthesized a high-purity, ultra-fine single perovskite $SrTiO_3$ powder as a photocatalyst for photochemical cells at low temperature using strontium nitrate and butyl titanate as raw materials using sol-gel technology and conducted a preliminary study on its influencing factors and basic characteristics. The results showed that ethanol, glacial acetic acid, water, glycerol, temperature and pH value had significant effects on the sol-gel process. Among them, the reaction temperature has the most obvious effect on the colloidal formation time and colloidal quality, and increasing the reaction temperature of the sol-gel process can greatly reduce the gelatinization time. However, too high a temperature will produce various forms of hydrolysis polymerization reactions, and at the same time crack the colloid, affecting the quality of the colloid. So the temperature is controlled at about 40°C. With the increase of heat treatment temperature, the crystal form of $SrTiO_3$ powder is more complete, the particle size and lattice constant increase, and the corresponding maximum absorption position of ultraviolet light is redshifted.

Chen Zhanfen [14] also mentioned the synthesis of sol gel method, $Co_3O_4/CdS/SrTiO_3$ ternary core-shell pn junction. Compared with $SrTiO_3$ nanospheres, the $Co_3O_4/CdS/SrTiO_3$ ternary core-shell pn junction exhibits excellent photocatalytic HER performance and cycling stability. The main reasons can be attributed to the enhanced response to visible light and the construction of ternary core-shell pn junctions. Among them, the narrow bandgap of CdS and Co_3O_4 can improve the utilization efficiency of visible light, thereby enhancing the utilization efficiency of solar energy. The construction of core-shell pn heterojunction can effectively excite and separate photogenerated carriers, promote the rapid migration of photogenerated electrons to water, and thus promote the generation of hydrogen gas. In addition, core-shell nanospheres with sufficient active sites are also an important reason for enhancing photocatalytic hydrogen production performance. This preparation method can effectively control the morphology and chemical properties of the final product.

Jiang Lei[15] also mentioned preparing a 10 mg · L^{-1} MB solution and continuing to dope Ag at the optimal W doping ratio. Exploring the effect of the proportion of W and Ag co doped $SrTiO_3$ on the degradation efficiency of MB, the degradation rate varies with the proportion of W and Ag co doped $SrTiO_3$. Compared with pure $SrTiO_3$, the photocatalytic activity of W and Ag co doped $SrTiO_3$ is greatly improved. As the proportion of Ag increases from 0.2% to 0.5%, the photocatalytic efficiency gradually increases. Continuing to increase the proportion of Ag doping leads to a decrease in photocatalytic activity. There is an optimal ratio for the co-doping of W and Ag in $SrTiO_3$, and distortion occurs inside the doped crystal, thereby improving the photocatalytic activity of $SrTiO_3$. The optimal photocatalytic performance is achieved when 2% W-0.5% Ag $SrTiO_3$ is doped.

Sol-gel method is a method to prepare nanometer $SrTiO_3$. Sol gel method has the characteristics of low reaction temperature, simple equipment, small and uniform particle size, high purity, etc., but there is certain agglomeration between particles and poor sintering performance. In the process of sol-gel reaction, how to control the hydrolysis and condensation rate plays a key role in obtaining uniform and fine particles. The industrial application of nanometer $SrTiO_3$ prepared by sol-gel method is difficult.

2.4. Chemical precipitation method

Liu Huanhuan [16] successfully prepared cubic $SrTiO_3$ by low-temperature direct precipitation method. In the process of preparing $SrTiO_3$ by low-temperature direct precipitation method, the effect of adding dispersants to the reaction system on the morphology and dispersibility of the powder particles was investigated. As the reaction time increases, the particle size growth of the product $SrTiO_3$ powder becomes larger. The influence of the law of reaction temperature on product particle size is that the higher the reaction temperature, the larger the particle size of $SrTiO_3$ can be obtained. The influence of initial liquid concentration on product particle size follows the following pattern: the higher the initial liquid concentration, the smaller the particle size of $SrTiO_3$ formed.

Wang Yanji [17] et al. investigated the effect of alkali addition on the quality of $SrTiO_3$ during the synthesis process, while keeping other conditions constant. As Sr/Ti gradually increases, the main content shows a trend of first increasing and then decreasing. The alkaline dosage between 145-155ml is ideal for the main content and Sr/Ti of the obtained product. The results indicate that within a certain range, as the amount of alkali increases, the amount of anhydrous ethanol increases, and the amount of water increases, the droplet acceleration of alkali solution and strontium nitrate solution slows down, resulting in an improvement in product quality. The main content of the product obtained under the condition of slow stirring of 4% sodium hydroxide with an ethanol addition of 75ml and a sodium hydroxide addition of 150ml is 99.71% strontium titanium ratio of 1.002.

The chemical precipitation method is to use a reagent as a chelating agent to precipitate strontium and titanium together under certain conditions; The precipitate is filtered, washed, and dried to obtain high-purity ultrafine $SrTiO_3$ powder. However, many substances are added during the synthesis of the powder, which can easily introduce impurities and reduce the quality of $SrTiO_3$ powder. And during the reaction process, agglomeration is prone to occur, making it difficult to control particle size. Therefore, research on dispersants is also important.

3. Environmental applications of nano $SrTiO_3$

Wu Weigang [18] studied the preparation of $SrTiO_3$ photocatalyst and its application in wastewater treatment. The photocatalytic results indicate that the oxygen vacancies on the surface of STO are the key factors affecting the spectral absorption and photocatalytic performance of this perovskite metal oxide. With the increase of oxygen defects on the surface of STO, the light absorption of STO samples in the visible and infrared regions is significantly enhanced, and an appropriate concentration of oxygen vacancies can improve the charge separation of photo generated carriers. The construction of oxygen defects on the surface can enhance the ability of $SrTiO_3$ to treat wastewater. Adopting appropriate and effective treatment methods to broaden the photo responsive range of the material while reducing the recombination rate of photo generated electrons and holes in nano SrTiO3, thereby improving its photocatalytic wastewater treatment ability, is currently the direction of research progress.

Ma Xuelian [19] studied the preparation and photocatalytic properties of perovskite type $SrTiO_3$ nano powder. The photocatalytic activity of $SrTiO_3$ catalyst prepared by carbon adsorption precipitation method and conventional precipitation method was evaluated using methylene blue aqueous solution as the target degradation product. $SrTiO_3$ precursors prepared by carbon adsorption precipitation method were calcined at 600 ℃, 700 ℃ and 800 ℃ for 2h respectively, and the catalyst nano powder corresponding to the calcination temperature was taken respectively. At room temperature, 20 mg of catalyst powder was added to 20 mL, 20 g/L methylene blue solution. The obtained suspension was continuously stirred in the dark for 20 minutes to achieve adsorption desorption equilibrium of methylene blue on the catalyst surface. Then put it into a self-made photoreactor, with a UV lamp (300 W, λ=365 nm, 10 cm away from the suspension) as the light source. Samples are taken every 10 minutes. The carbon adsorption precipitation method can effectively reduce particle aggregation and sintering during high temperature calcination and suppress particle growth. It has a good photocatalytic effect on methylene blue, and within 60 minutes, methylene blue is almost completely decomposed on the $SrTiO_3$ catalyst.

Ren Jinqiu's research on the synthesis and treatment of simulated [20] radioactive wastewater using aluminum doped $SrTiO_3$ photocatalyst mentioned that the synthesized $SrTiO_3$ is mostly cubic, partially amorphous, and contains some rutile TiO_2. After modification with Al element, the morphology of the synthesized sample becomes more regular, with some exhibiting flakes like morphology. According to UV-VIS-DRS testing and Taut formula calculation, the energy gaps of the three synthesized samples were found to be 3.68 eV, 3.76 eV, 3.80 eV, and 3.88 eV, respectively. In terms of photocatalytic reduction of toxic metals, hexavalent chromium was used as the pollution source, and inexpensive commercial P25 was used as the photocatalyst. Various reaction conditions were adjusted for experiments to determine the optimal conditions for photocatalytic reaction. The result showed that acidic conditions were more conducive to the reaction. When the alkalinity is too high, Cr (VI) is reduced to Cr (III) and easily combines with OH - in the solution, covering the surface of the photocatalyst and hindering the progress of the reaction. The addition of sacrificial agent methanol accelerates the reaction, but its dosage cannot exceed 0.4 mg/L, otherwise the oxidation-reduction reaction between methanol and Cr (VI) will be the dominant factor.

The photocatalytic performance of semiconductor $SrTiO_3$ in wastewater treatment is affected by its high recombination rate of photogenerated electron hole pairs and narrow photo response range caused by its large bandgap. Therefore, research on the modification of the photocatalytic performance of $SrTiO_3$ is very important. The application of nano $SrTiO_3$ in actual domestic sewage treatment still needs a long way to go. Especially in the treatment of nuclear wastewater, it is still in its infancy.

4. Conclusion

Nano $SrTiO_3$ is a highly promising photocatalyst. This article extensively investigates its synthesis progress and application aspects, and draws the following conclusions:

(1) The hydrothermal method can directly prepare nano $SrTiO_3$ powder from the liquid phase, with low synthesis temperature, low production cost, and high product quality, but with long reaction time and relatively high equipment requirements. The prepared nano $SrTiO_3$ has high purity, uniform particle size, controllable morphology and crystal surface.

(2) High temperature solid-phase reaction method is a traditional method for preparing nano $SrTiO_3$ powder. The high-temperature solid-state reaction method has a simple process, short reaction time, and easy control of operation, but the product is prone to agglomeration, uneven particle size, and high process energy consumption. The low-temperature solid-phase reaction method has the characteristics of short reaction time, high yield, low energy consumption, and simple process. The low-temperature solid-phase reaction method does not use solvents and has low environmental pollution, making it suitable for large-scale production and providing technical support for the industrial application of $SrTiO_3$.

(3) Sol gel method has the characteristics of low reaction temperature, simple equipment, small and uniform particle size, high purity, etc., but there is certain agglomeration between particles and poor sintering performance. In the process of sol-gel reaction, how to control the hydrolysis and condensation rate plays a key role in obtaining uniform and fine particles.

(4) The chemical co-precipitation method is another way to obtain high-purity ultrafine $SrTiO_3$ powder. However, many other substances are added during the synthesis process, which can easily introduce impurities and reduce the quality of $SrTiO_3$ powder. And during the reaction process, agglomeration is prone to occur, making it difficult to control particle size.

(5) Due to its excellent photocatalytic activity, nano $SrTiO_3$ has good degradation performance for heavy metal ions and organic pollutants in wastewater. However, there is still a long way to go in practical application of domestic wastewater treatment, and it is still in its infancy ecological wastewater treatment.

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