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Preparation and Application of Strontium Titanate/Doped Strontium Titanate Single Crystals

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Abstract: Strontium titanate (SrTiO₃) has excellent electrical and optical characteristics, which have made it a very popular material in high-k capacitors, oxygen sensors, superconducting materials, photocatalysts, and resistive random access memory (RRAM). This paper provides a review of the preparation and application research of SrTiO₃ and the doped single crystals with a systematic analysis of the common preparation paths which include solid-phase, sol-gel and single crystal growth. The solid-phase technique has advantages of ease and scalability, whereas the sol-gel technique permits to refine the grain and achieve compositional uniformity. Moreover, doping with elements like La⁺, Nb5⁺, Fe⁺, Co²⁺ and Mn⁺ can efficiently be used to manipulate its band structure. The experimental observations indicate that as the sintering temperature rises the crystallinity of the sample is rising and the grain size is growing as at 800degC and 1100degC, the thickness of the grain is 75 nm and 180 nm, respectively. Doped SrTiO₃ has a smaller bandgap and a higher photocatalytic performance, and its wide application opportunities in the environmental cleanup and photocatalytic systems, optoelectronics, and energy conversion are evident.

1. Introduction

Strontium titanate (SrTiO₃), which is a common perovskite oxide compound, has emerged as a prime research material in the field of multifunctional materials, as it displays great electrical, optical and chemical stability. SrTiO₃ has found extensive application in high-k capacitors, oxygen sensors, thermoelectric materials, photocatalysts, and resistive random access memory (RRAM) because it has a large dielectric constant, low dielectric loss, good thermal stability, and versatile band structure since it was first discovered in the mid-20th century. Specifically, SrTiO₃, as one of the n-type semiconductors with a broad bandgap of about 3.2 eV, can be useful in decomposing water to generate hydrogen or degrading organic pollutants under the ultraviolet light in photocatalysis and photoelectric conversion, thus showing high potential in environmental and

energy applications.

Hence, in terms of material preparation and performance control, this article will systemicly survey the preparation procedures, structural features and application developments of strontium titanate and its doped single crystal with an eye to the examination of the impact of the doping on the band structure and optoelectronic characteristics of the materials as theoretical reference and experimental foundation of the future material planning and optimization of the functionality toward the objectives.

2. Related Work

In recent years, perovskite oxides have become a hot research topic in photocatalysis, electronics, and optoelectronics due to their excellent optoelectronic properties, tunable band structures, and multifunctional application potential. The following reviews the latest research progress in SrTiO₃ and its doped systems in photocatalysis, dielectric properties, magnetoelectric properties, and thin-film devices.

Assavachin et al. quantitatively evaluated the charge separation ability of (100), (110) and (111) crystal planes in photocatalytic water splitting by hydrogen annealing SrTiO₃-x single crystals. The results showed that different crystal planes exhibited different water oxidation photocurrents, voltages and surface photovoltages due to differences in work function, and the electron transfer barriers were (100) < (111) < (110) [1]. Zhu et al. added 0.5-2% ammonium tetrathiomolybdate precursor to SrTi0.85Nb0.15O3 ceramics. After sintering at 1700 K in hydrogen/argon, the precursors were converted into MoS2, MoOx and Mo particles in sequence, promoting grain boundary improvement and secondary phase formation [2]. Mohan et al. prepared Sm and Ag doped SrTiO3 nanoparticles by sol-gel method for photocatalytic degradation of methyl blue under visible light. Doping reduces particle size and lowers the band gap. Sm@Ag@SrTiO₃ has the highest photocatalytic activity, and the MB (Methylene Blue) degradation rate reaches 80% [3]. Aly et al. prepared La₂O₃ doped BNKT-ST ceramics by solid phase method and systematically studied the effect of La doping on phase structure, dielectric properties, ferroelectricity, piezoelectric response and energy storage density. The results showed that high La concentration can reduce the grain size. The piezoelectric constant d33 of the 0.020 mol% doped sample reached 121 pc/N, the energy storage density of 0.97BNKT-0.030ST was 0.26 J/cm³, and the efficiency was 58% [4]. Hashim et al. prepared PMMA-PS composite films doped with SrTiO₃ nanoparticles for use in optical and optoelectronic devices. The study showed that when the SrTiO₃ content reached 5 wt.%, the film absorption was enhanced, the transmittance was reduced, the band gap was reduced, and the optical constants (absorption coefficient, refractive index, extinction coefficient, dielectric constant and optical conductivity) increased [5]. Mohammed and Ghaleb used the DFT (Density Functional Theory) method to systematically analyze the structural, electrical and optical properties of cubic perovskite SrTiO₃, using LDA (Local Density Approximation), GGA (Generalized Gradient Approximation) and M-GGA (Meta-Generalized Gradient Approximation) functionals, and compared them with experimental data. The results showed that M-GGA (Meta-Generalized Gradient Approximation) was closest to the experiment in terms of lattice parameters, band structure and optical absorption edge, revealing the characteristics of Ti-O hybridization and Ti 3d t2g-eg splitting [6]. Alghamdi et al. prepared (1-y) Co_{0.8}Cu_{0.2}Fe₂O₄ + y SrTiO₃ magnetoelectric nanocomposite materials and studied their mechanical and radiation shielding properties. Compared with standard materials, it was shown that SrTiO₃ doped nanoferrite has good application potential in ionizing radiation protection [7]. Xu et al. prepared Rh and C-doped SrTiO₃ cubic perovskite by solid-phase method for tribocatalytic dye degradation. Doping introduced lattice contraction, stress and oxygen vacancies, significantly improving the tribocatalytic efficiency. The degradation rate of

Rh-doped sample for rhodamine B reached about 88%, and the reaction rate constant was 0.9 h⁻¹[8]. Akhtar et al. prepared low-level Zr-doped (Ba_{0.9}Sr_{0.1})Ti_{1-x}ZrxO₃ ceramics (x=0–0.04) by solid-phase method. XRD (X-ray Diffraction) showed a stable pseudo-cubic perovskite phase, and SEM showed that the grains were uniform and decreased with the Zr content [9]. Karmakar et al. first grew single crystal ZnGa₂O₄(111) thin films on Nb-doped SrTiO₃ substrates by pulsed laser deposition for MOS (Metal-Oxide-Semiconductor) capacitor research. XRD/XPS (X-ray Photoelectron Spectroscopy) confirmed the single crystal structure, and ultraviolet absorption measured an ultra-wide band gap of approximately 5.02 eV, showing high thermal stability and low leakage current. High-temperature current-voltage and capacitance measurements showed that the Poole-Frankel mechanism dominated the medium- and high-field current, and the Schottky mechanism dominated the low-field current. The interface and oxide charge increased with temperature, and the dielectric constant decreased. The film showed excellent performance and reliability in high-temperature environments [10]. Photocatalytic degradation has attracted attention as a green and efficient strategy. Among them, new perovskites have shown potential in the degradation of dyes, drugs and industrial waste due to their unique structure, excellent photoelectric properties, tunable band gap and efficient carrier transport. Terhemba et al. reviewed its sustainable synthesis methods, environmental applications and challenges [11]. Although SrTiO₃ and its doping systems have shown excellent performance in photocatalytic, electrical and optoelectronic devices, existing research generally has bottlenecks such as difficulty in controlling doping uniformity, crystal defects affecting performance, and immature large-scale controllable preparation technology.

3. Method

3.1 Solid-phase Method

Strontium titanate (SrTiO₃) is a widely used functional material with high dielectric constant, low dielectric loss, good thermal stability, a wide bandgap of approximately 3.2 eV, and excellent photocatalytic properties. It has important applications in high-dielectric-constant devices, oxygen sensors, superconductors, photocatalytic materials, and resistive random access memory. Its single crystals can also be used as optical materials and artificial gemstones, such as in optical components, lenses, and artificial gemstones. Furthermore, srtio₃ single crystals are excellent substrate materials for high-temperature superconducting thin films and various oxide thin films.

Solid-state crystal growth (SSCG) is being explored as a solution to several current technical challenges in metal oxide preparation. Long-standing production methods have been plagued by challenges such as high-temperature heating, maintaining raw material consistency, and controlling impurities. However, these traditional methods are subject to minor defects, resulting in low efficiency. Continuous improvements in heat treatment and heating processes are enabling the mass production of high-quality, reproducible, and structurally stable products, with the potential for long-term, controllable preparation processes. Solid-state crystal synthesis is considered a sustainable technology, offering numerous advantages in terms of efficiency and simplicity. This method is typically performed in a high-temperature furnace and does not require additional complex equipment. However, long-term continuous preparation remains challenging due to factors such as uneven chemical composition, varying elemental reactivity, melting behavior, and volatilization of some elements. However, compositional uniformity, crystal integrity, and stability under constant temperature are crucial for solid-state synthesis products. While its performance is relatively limited, it facilitates achieving uniform compositional distribution. This method is expected to remain an important preparation technique for a wide range of oxide systems. Once a primary crystal structure is achieved through SSCG, it can be further processed to produce larger or higher-purity crystals.

Perovskite-based semiconductor materials have gradually attracted the attention of researchers due to their advantages such as long lifetime of photogenerated carriers, high chemical and thermal stability, and strong catalytic activity. Strontium titanate (SrTiO₃), as a typical multi-metal oxide, has been widely used in the field of photoelectrocatalysis. However, there are relatively few studies on the synthesis of SrTiO₃ photocatalysts and their photocatalytic water splitting. SrTiO₃ nanoparticles obtained by different preparation methods have obvious differences in morphology, uniformity and particle size. Photocatalytic activity and material stability are the key factors limiting the further improvement of the photocatalytic performance of SrTiO₃.

SrTiO₃has a typical perovskite structure, combining advantages such as high energy efficiency, low power consumption, and good thermal stability. It is one of the most promising inorganic oxides in the fields of ceramics, capacitors, gas sensors, and functional thin film materials. With the growing demand for optoelectronic functional materials, SrTiO₃ has been widely used due to its high purity, controllable particle size, uniform distribution, and strong reactivity. In recent years, its preparation in various structural systems (such as linear, tubular, and porous structures) has been intensively studied. SrTiO₃ materials show high preparation flexibility and high yield. However, in most synthesis processes, its morphology regulation, dispersion control, and performance maintenance still face certain difficulties. Related studies have further improved the photocatalytic performance and structural stability of SrTiO₃ by improving the heat treatment system, adjusting the reaction conditions, and optimizing the morphology control model.

3.2 Sol-Gel Method

The sol-gel method is a wet chemical preparation technology that typically uses metal alkoxides or inorganic salts as precursors to generate a sol through hydrolysis and polycondensation in an organic solvent or aqueous phase. The sol is then converted into a gel through aging and drying, and then subjected to high-temperature heat treatment to obtain the desired material. The hydrolytic sol-gel method is commonly used, and its core process includes the hydrolysis reaction of the precursor and the polycondensation reaction of the hydrolysis product. Taking a metal alkoxide M(OR) (M represents a metal element and R is an alkyl group) as an example, its hydrolysis reaction can be expressed as:

$$M(OR)_n + nH_2O \rightarrow M(OH)_n + nROH$$

 $M(OH)_n$ is the metal hydroxyl compound generated by hydrolysis, and ROH is the corresponding alcohol.

Condensation reactions include dehydration polycondensation and alcohol dehydration polycondensation; such as dehydration polycondensation:

Alcohol loss polycondensation:

By controlling the rates and conditions of the hydrolysis and polycondensation reactions, the structure and properties of sols and gels can be manipulated. However, raw materials such as metal alkoxides are often expensive, and the reaction requires high purity of the organic solvent. Consequently, the non-hydrolytic sol-gel method has been developed. Water-sensitive materials (such as T and Zr) can be prepared using this method. Sols and gels are primarily generated through reactions between metal halides and organometallic compounds or other organic reagents (such as ethers and ketones).

Appropriate organometallic compounds or inorganic salts, such as titanium acetate, isopropyl titanate, strontium carbonate, or strontium nitrate, are selected as Sr and Ti precursors and dissolved in an organic solvent or aqueous solution. A complexing agent (such as ethanol, acetic acid, or citric acid) is added to stabilize the solution. This step allows for control of solution homogeneity and reaction rate by adjusting the pH, solvent type, and concentration, providing a stable precursor system for subsequent gelation. The precursor undergoes hydrolysis and polycondensation reactions under stirring to form a uniform sol. The hydrolysis reaction converts the organometallic compound into a metal hydroxyl group, while the polycondensation reaction gradually forms a metal-oxygen bond network. By controlling the hydrolysis rate, solvent ratio, and reaction temperature, the viscosity, particle size, and dispersibility of the sol can be effectively adjusted, thereby influencing the microstructure of the final material. The dried gel is calcined at high temperature (typically 600–1000°C) to convert the amorphous oxide into crystalline SrTiO₃. During calcination, the particles sinter and grow, reducing lattice defects and improving the material's crystallinity and performance.

3.3 Preparation and Performance Control of Doped Strontium Titanate Single Crystals

Doped strontium titanate (STO) single crystals can be effectively tuned by introducing appropriate amounts of metal ions or non-metallic elements. Common doping elements include La³⁺, Nb⁵⁺, Fe³⁺, Co²⁺, and Mn³⁺. Different doping ions can introduce additional carriers, manipulate the band structure, or improve lattice defects, thereby enhancing the material's performance in applications such as photoelectrocatalysis, gas sensing, and functional thin films.

In the solid-phase method, the doping source is evenly mixed with the SrTiO₃ precursor, and then sintered at high temperature to generate a doping precursor powder, which is then grown to obtain a single crystal. This method is simple to operate and low in cost, but the distribution uniformity of the doping elements and the integrity of the crystal are difficult to control. The sol-gel method uses a chemical solution to achieve uniform dispersion of doping ions. After drying, gelation and high-temperature calcination, a doped powder is formed, which is further used for single crystal growth, effectively improving the composition uniformity and microstructure control. For high-purity and high-crystallinity single crystals, the Czochralski method and the floating zone method are widely used. By precisely controlling the melt temperature, crystal pulling rate and atmosphere conditions, single crystals with larger size, uniform doping and fewer defects can be obtained.

4. Results and Discussion

4.1 Materials and Reagents

Strontium precursors used were SrCO₃, Sr(NO₃)₂, or strontium carbonate. Titanium precursors included Titanium isopropylate (TTIP), Titanium tetraethoxide (TEOT), or TiO₂ nanopowder. Dopant sources comprised Nb₂O₅, NbCl₅, La₂O₃, Sm₂O₃, AgNO₃, RhCl₃, ZrO₂, and others. Solvents employed were ethanol, isopropyl alcohol, deionized water, and acetic acid. Complexing agents and stabilizers included citric acid, acetic acid, and ethylene glycol. Flux and crystal growth agents consisted of NaCl, KCl, LiF, and MoO₃, depending on the specific protocol. Reactions were carried out under atmospheres of air, argon, or H₂/Ar mixtures for reduction treatment. Photocatalytic test reagents included Methylene Blue (MB), Rhodamine B (RhB), isopropyl alcohol (•OH scavenger), and Na₂S/Na₂SO₃ as sacrificial reagents.

4.2 Preparation Plan

A. Powder Preparation

A1. Solid-Phase Method (for ceramic/ceramic samples)

Mixture ratio: Sr:Ti = 1:1, doping at the target molar ratio (e.g., Nb 0.5–5 at.%).

Weigh and mix thoroughly, then ball mill (or mortar) for 6–12 hours (using isopropyl alcohol as the medium).

Pre-sintering: 900–1000 °C in air, hold for 4–6 hours, then cool and grind.

Secondary sintering: 1200-1400 °C, hold for 6-12 hours; H2/Ar reduction treatment (500-800 °C, 1-4 hours) is optional.

Grind and sieve to obtain the final powder.

Recommended parameters: Ball milling for 6–12 hours; Pre-sintering at $1000 \, \text{C}$ for 4 hours; Calcination at $1300 \, \text{C}$ for 6 hours; Reduction in H2/Ar at $650 \, \text{C}$ for 2 hours. A2. Sol-Gel Method (for Nanostructured/Thin Films)

Dissolve TTIP in anhydrous isopropanol and add acetic acid; dissolve the Sr precursor in ethanol/water or form a dispersion.

Slowly add the sr solution dropwise to the ti solution and stir for 2–6 hours, maintaining a ph of \approx 2–5, to obtain a uniform sol.

Age the solution for 12–24 hours and dry at 80–120 °c to obtain a gel.

Preheat at $300-500\,^{\circ}$ c to decompose organic matter, then calcine at $600-1000\,^{\circ}$ c for crystallization (2–6 hours).

Thin films: spin coating followed by layered heat treatment.

Recommended precursor concentration: 0.2-0.5 m; dry the gel at 100 °c for 12 hours; calcine at 800 °c for 2 hours.

B. Single crystal growth

B1. molten salt solution method (low-temperature crystal growth)

Mix srtio3 powder with molten salt (e.g., nacl-kcl) and place in a crucible.

Heat to the melting point of the molten salt (600–900 °c), maintain temperature to promote growth, and slowly cool (0.1–1 °c/h).

Dissolve the molten salt, clean, and extract the crystal.

Suitable for small, high-quality crystals.

B2. floating zone method / optical melt pulling (high-purity single crystals)

Isostatically press the high-purity powder into a rod and sinter at high temperature (≥1400°c).

Floating zone growth: growth rate 1–10 mm/h, rotation speed 10–30 rpm, controlled atmosphere (oxygen/argon/vacuum).

Post-annealing (oxygen 800–1000 °c) to eliminate surface oxygen defects (if necessary).

Advantages: produces large, highly crystalline single crystals; disadvantages: high equipment requirements.

Doping note: the doping amount is usually 0.1–5 at.%; first fully homogenize it at the powder stage (high temperature calcination + ball milling cycle).

4.3 Sample Naming and Control Design

S0: Pure SrTiO₃ (baseline)

Snb_0.5: Nb-doped 0.5 at% (Nb) (snb_1, snb_5)

Sla_1: La-doped 1 at% (La) (sag_1, srh_0.5, etc.)

Sred: H2-reduced sample

4.4 Data Analysis

As shown in Table 1, the average particle size and crystallinity of the strontium titanate samples show a significant upward trend with increasing calcination temperature. Sample S1 exhibited a particle size of only 75 nm and a crystallinity of 82.5% at 800 $^{\circ}$ C, indicating that the grains had not fully grown at this low temperature and that the lattice contained some defects. Upon increasing the temperature to 900 $^{\circ}$ C (sample S2), the particle size increased to 90 nm and the crystallinity rose to 88.3%, indicating that some amorphous regions began to transform into an ordered lattice structure. When the calcination temperature reached 1000 $^{\circ}$ C (sample S3), the grains further grew to 120 nm, and the crystallinity reached 93.7%, indicating a more complete crystalline structure. Further increasing the temperature to 1100 $^{\circ}$ C (sample S4), the particle size rapidly increased to 180 nm, and the crystallinity increased to 95.2%. At this point, intergranular sintering was enhanced, and grain boundaries gradually decreased.

,	Table 1 Particle size and crystallinity of solid phase method samples					
<u> </u>	Calcination Temperature (°C)	Average Particle Size (nm)	Crystallin			

Sample ID	Calcination Temperature ($^{\circ}$ C)	Average Particle Size (nm)	Crystallinity (%)
S1	800	75	82.5
S2	900	90	88.3
S 3	1000	120	93.7
S4	1100	180	95.2

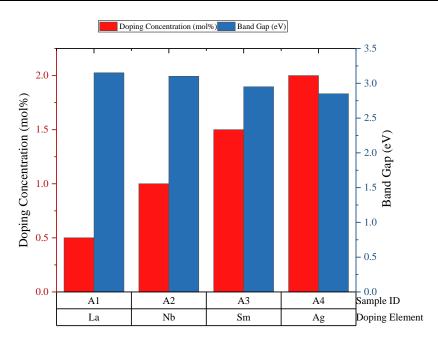


Figure 1 Doping elements and energy band width

The energy band width of undoped SrTiO₃ is about 3.2 eV, but the band gap shrinkage phenomenon is generally observed after doping. Among them, the energy band width drops to 3.15 eV when La doping is 0.5 mol%, indicating that a small amount of rare earth elements can introduce shallow energy levels and slightly improve the light absorption performance. When the doping concentration is further increased and different elements are used, the energy band width decreases more significantly. For example, when Sm doping is 1.5 mol%, the band gap decreases to 2.95 eV, while when Ag doping is 2.0 mol%, the lowest is only 2.85 eV. Figure 1 shows that doping

with transition metals and precious metal elements can introduce impurity energy levels in the SrTiO₃ lattice, effectively narrowing the band gap, thereby improving its response to visible light.

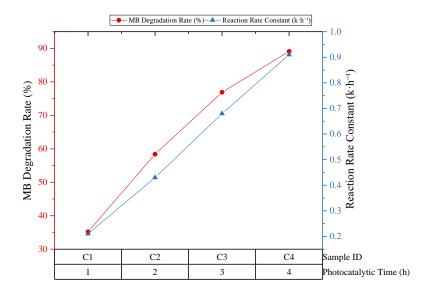


Figure 2 Photocatalytic performance test results

Within one hour of reaction, the MB degradation rate of sample C1 was only 35.2%, with a reaction rate constant of 0.21 h⁻¹, indicating that the active sites were not fully utilized in the initial stage. As the illumination time increased to 2 and 3 hours, the degradation rate increased to 58.4% and 76.9%, respectively, with corresponding reaction rate constants of 0.43 and 0.68 h⁻¹, reflecting a significant increase in the efficiency of photogenerated electron-hole pair separation. When the reaction time was extended to 4 hours, the degradation rate of sample C4 further increased to 89.1%, with the reaction rate constant reaching a maximum value of 0.91 h⁻¹, indicating that the system had reached the reaction saturation stage (Figure 2).

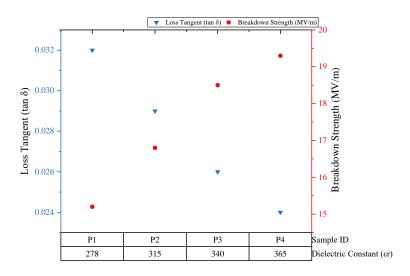


Figure 3 Dielectric performance parameters

Sample P1 has a dielectric constant of 278, a loss angle of 0.032, and a breakdown strength of 15.2 MV/m, indicating a weak dielectric response and high energy loss in the initial state. With the optimization of the sample structure and grain distribution, the dielectric constants of samples P2

and P3 increased to 315 and 340, respectively, while the loss angle decreased to 0.029 and 0.026, indicating that the internal polarization response of the material was enhanced and the dielectric loss was effectively suppressed. When the sample was further optimized to P4, the dielectric constant reached a maximum value of 365, the loss angle dropped to a minimum of 0.024, and the breakdown strength also increased to 19.3 MV/m, demonstrating excellent comprehensive dielectric properties, as shown in Figure 3.

5. Conclusion

This paper has made a systematic study of the preparation techniques and the performance optimization concepts of strontium titanate (SrTiO₃) and doped single crystals. A comparison and analysis of the various processes (solid-phase, sol-gel, and floating-zone) were performed to verify the important role of the process parameters on the grain size, crystallinity and defect concentration. Doped samples in photocatalytic experiments exhibited increased efficiency and stability in the visible light response range in photogenerated carrier separation indicating that the doping approach is effective in bandgap control. Moreover, a correlation model between process, structure, and performance was created with the help of microstructural characterization and performance testing, which offered an experimental foundation of functional design of related materials. The concentration range of the doped samples in the experiment was narrow and not in the systemic coverage of the most appropriate ratios of different elements. Also the change of electronic structure can be calculated and analyzed theoretically; this needs more research. In the future, first-principles calculations can be combined with in situ characterization methods to gain additional understanding of the underlying processes or mechanisms of doping-based bandgap manipulation and defect state distribution.

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