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Study on Preparation Technology of Transition Metal Sulfide Nanomaterials

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Abstract. Transition metal sulfide nanomaterials (nano TMS), with their unique size, surface and quantum effects, have been playing an increasingly important role in industries, such as semiconductors. However, the properties of nano TMS are still under investigation. In order to promote their widespread application, it has been a critical issue for material preparation technology to develop a proper processing method. This article summarizes common methods for preparing nano-TMS, including top-down preparation methods using mechanical or liquid ultrasonic exfoliation, bottom-up preparation methods involving hydrothermal and solvothermal methods, chemical vapor deposition, and electrochemical deposition (collectively known as bottom-up preparation methods), as well as biomineralization production based on sulfate-reducing bacteria metabolism. It also provides examples and corresponding selection criteria. Additionally, this article analyzes the existing shortcomings in nano-TMS preparation from four aspects: production stability, detection ways, cost control, and environmental damages, and identifies potential development directions such as high-throughput microreactor technology, more research on biomineralization mechanisms, and AI-assisted reaction conditions control. It offers valuable references for large-scale industrial production and application of nano TMS.

Keywords: Nanomaterials, transition metal sulfide, thin film materials, semiconductor

1. Introduction

Metal sulfides are compounds composed of a single or multiple metal elements and sulfur. Based on the classification and number of metal elements, they can be categorized into three types: main-group metal sulfides, transition metal sulfides, and poly-metallic sulfides. Main-group metal sulfides are commonly used in organic synthesis and solar cells, as well as thermoelectric materials. In contrast, transition metal sulfides (nano TMS) have even broader applications, with more diverse preparation methods. This article primarily discusses the preparation techniques for transition metal sulfides (hereinafter referred to as nano TMS).

The basic unit of nano TMS is a cluster MS_x formed by the combination of transition metal M and sulfur S. Its structure is diverse, possibly in planar layered (MoS_2), wurtzite-type (ZnS), or spinel-type (Co_3S_4), but at least one dimension has a nanoscale range (0-100 nm). According to the dimensions present at non-nanoscale (i.e., macroscopic) scales, they can be classified as quantum dots (all three dimensions are nanoscale), nanowires, nanosheets, and porous bodies. They possess numerous unique physical properties, such as surface effects due to extremely high specific surface area, optoelectronic

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characteristics dependent on quantum dot size (quantum confinement effect and bandgap modulation effect), and the ability to absorb light and efficiently convert it into thermal energy.

Nanotextured TMS has an extremely broad range of applications: utilizing its numerous pores and large interlayer spacing, it possesses strong energy storage capabilities. The CuS/Sn S_2 used as the battery anode can lead to increased energy density, with Coulombic efficiency exceeding 99.5% [1]; leveraging its bandgap modulation and quantum confinement effect, it can achieve photovoltaic conversion, such as CdS and ZnS forming the core of solar cells, and high-purity quantum dots for high NTSC color gamut quantum dot TVs [2]; in addition to these applications in semiconductors, nanotextured TMS, due to its porous structure, large specific surface area, and abundant electronic and orbital states in transition element atoms, can also serve as excellent catalytic materials and gas capture and detection materials. Furthermore, based on its plasma resonance effect, nanotextured TMS can be used for photothermal therapy [3], among other applications.

In the foreseeable future, nanomaterials, with nano-TMS as a key representative, will become one of the most important industrial pillars. However, the preparation of nano-TMS is currently focused on miniaturization and experimental production, making large-scale manufacturing difficult. To promote its scaled-up production, the preparation of nano-TMS has become an increasingly important topic.

2. Preparation technology and case of nano-TMS

According to the preparation scale and raw materials of nano-TMS, its preparation is generally divided into two types: "top-down" and "bottom-up." The former starts from larger sizes, gradually thinning down through methods such as exfoliation and etching until the thickness reaches the nanometer level. This method is more suitable for creating nano-TMS with regular planar shapes; the latter begins with smaller sizes, forming smaller nano-TMS particles through chemical or physical means, which are then deposited and grown into specific patterned nano-TMS materials using templates. This method is more versatile in terms of the dimensions and forms of nano-TMS materials.

2.1. Top-down methods—stripping

The exfoliation method is a crucial approach for preparing two-dimensional nanotransmission line materials. Its core principle involves using physical or chemical means to overcome interlayer van der Waals forces, thereby separating bulk materials into single-layer materials at the nanoscale. This method is applicable to transition metal sulfides with layered stacking structures, such as layered Mo S_2 . The crystal structure is shown in Figure 1.

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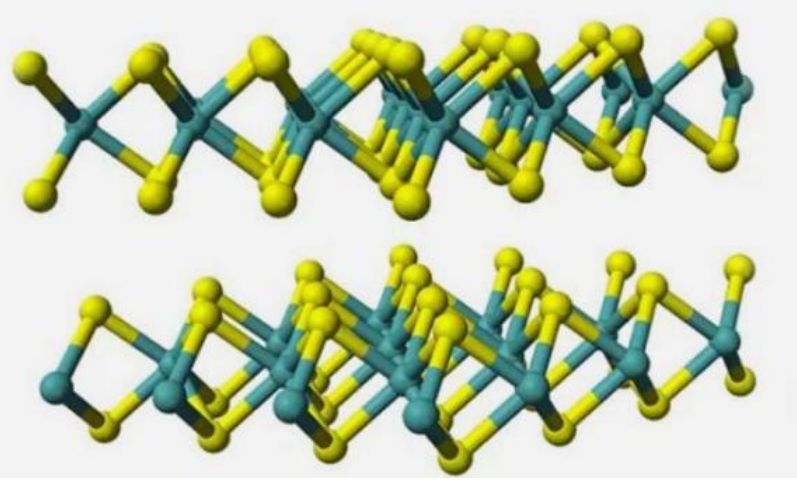


Figure 1: The layered structure of MoS_2 (with Mo in the middle and S atoms at the top and bottom)

In the exfoliation method, the most commonly used techniques are mechanical exfoliation and ionic intercalation exfoliation. Mechanical exfoliation involves using mechanical force to overcome interlayer van der Waals forces, including the tape exfoliation method, where strong adhesive tape is repeatedly applied and removed, and the liquid ultrasonic exfoliation (LPE) method, which uses the wetting effect of liquids to increase interlayer spacing under ultrasonic shear forces, thereby weakening van der Waals forces [4]. Ionic intercalation exfoliation achieves exfoliation by introducing ions (such as commonly used ones Li^+ and Na^+) into the interlayers. When some metals generate ions, they react with water to produce H_2 gas, further increasing the interlayer spacing.

Mehta et al. used blue light tape to mechanically exfoliate MoS_2 . They first peeled off thin sheets from the bulk MoS_2 crystal using a layer of blue light tape, and then transformed these into few-atom layers through repeated peeling on the tape, and transferred them onto pre-treated Si/ SiO_2crystals, further reducing their size to nanoscale. The team then doped the films in 1,2-dichloroethane and ozone environments to study their current response to light under different treatments [5].

Tang prepared MoS_2 nanosheets using liquid-phase ultrasonic exfoliation. A molybdenite sample was firstly polished to nearly 2mm using a planetary mill, and then it was sieved through a 45μm screen and treated with an ultrasonic homogenizer in a 45% ethanol solution at 500W for 12h. Afterwards, the suspension was centrifuged at 5000 r/min for half an hour, and the supernatant was collected as the top 60% solution. It yielded a liquid containing dispersed MoS_2 nanosheets. The team then used static gas mixing to study the gas-sensitive properties of the nanosheets towards ammonia and proposed a method for measuring ammonia using MoS_2 nanosheets [6].

Yang prepared MoS_2 nanosheets with the assistance of lithium-ion intercalation and exfoliation. They mixed MoS_2 powder with n-butyl lithium in n-hexane and stirred the components at room temperature under nitrogen protection for 48 hours. Afterwards, it underwent 3-4 centrifugal precipitations at 10,000 rpm to remove remaining n-butyl lithium. Then the unexfoliated material was exfoliated at 600W power for an hour, followed by centrifugation to separate the incompletely exfoliated precipitates. The obtained MoS_2 nanosheets were processed into flexible thermoelectric thin films using vacuum filtration and various Lewis acids, and their thermoelectric properties were measured [7].

Three different exfoliation methods each have their own advantages and disadvantages: mechanical exfoliation is the simplest but has the lowest yield and is only suitable for small-scale production; liquid ultrasonic exfoliation and ion intercalation exfoliation can significantly improve production efficiency

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but are more complex and may lead to impurities, defects, or phase transitions in materials (for example, lithium-ion ultrasonic exfoliation can cause Mo S_2 materials to transition from 2H phase to 1T). A common problem for all exfoliation methods is low control accuracy, making it difficult to precisely control the size and structure of the generated nanosheets. So they may not be suitable for high-precision production.

2.2. Bottom-up methods

Unlike the top-down methods, the bottom-up preparation method involves the gradual accumulation of atoms, ions, and clusters from few to many, ultimately forming specific nano-TMS materials. The most commonly used methods are deposition techniques, including hydrothermal and solvothermal reactions based on liquid-phase chemistry (including the improved microwave hydrothermal method), chemical vapor deposition (CVD) based on gas-phase chemistry, and electrochemical deposition based on electrochemical reactions.

2.2.1. Hydrothermal and solvothermal methods

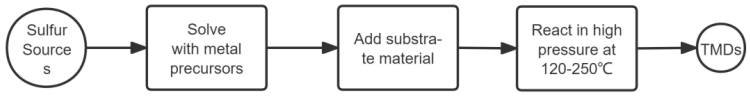


Figure 2: Basic process of hydrothermal and solvothermal methods

The basic process of hydrothermal and solvothermal methods is shown in Figure 2. Sulfur sources (such as soluble sulfides or small organic molecules containing sulfur) are thoroughly dissolved in a solvent along with metal precursors (mostly soluble inorganic or organic salts containing relevant metals). A substrate material with the same shape as the target material is then added to control its morphology. Then the mixture is heated and pressurized at 120-250 ℃ to form nanomaterials with specific shapes. Sometimes, microwaves can be introduced to accelerate the reaction rate.

Li et al. used nickel foam (NF) as the metal precursor and thiourea as the sulfur source to prepare porous Ni S_x @NF catalysts. The 1*3 cm^2 nickel foam was treated in sulfuric acid for 5 minutes and rinsed with ethanol and deionized water, then dried overnight at 60 ℃. Thiourea of different masses (0.07g, 0.14g and 0.28g) was mixed with the nickel foam, and then the mixture reacted in a polytetrafluoroethylene reactor at 150 ℃ for about 5 hours. After filtrating and drying, the Ni S_x@NF catalysts were obtained. The team then characterized the catalysts and compared their HER (hydrogen evolution reaction) performance in simulated seawater [8].

Similarly, Mo et al. used the ethylenediamine-anhydrous ethanol solvothermal method, with sublimed sulfur as the sulfur source and ultrasonically treated nickel-iron foam as the nickel precursor. After thoroughly mixing the sulfur source solution with the nickel precursor, they reacted in a vacuum drying oven at 180 ℃ for about 18 hours. The mixture was then cooled to room temperature, washed, and dried to obtain NiFeS nanosheets. These nanosheets exhibit high electrical conductivity and abundant active sites, making them a low-cost, efficient, and highly active HER catalyst [9].

Chen processed AgIn S_2 (AIS) quantum dots using microwave-assisted hydrothermal synthesis. They used AgN O_3 , and In $(NO_3)_3$ ·4.5 H_2 O as Ag and In precursors and sodium sulfide as the sulfur source. After mixing the Ag and In precursor solutions in a ratio of Ag:In = 0.11 to 0.25, they added organic ligand to balance cationic reactivity, diluted the solution, and added BmimB F_4 to enhance its wave absorbance. With a pH of about 10, they added the sulfur source and stirred for 10 minutes. The mixed solution was then transferred to a microwave synthesizer, where it reacted at 100-130 ℃ for no

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more than 25 minutes to obtain the quantum dot dispersion. The dispersion was centrifuged in isopropanol, purified, and dried to yield the quantum dots. At last, the group used the same method to coat ZnS on the surface of AIS quantum dots, further enhancing their fluorescence performance [10].

Hydrothermal and solvothermal methods are among the key approaches for preparing nano TMS. Their characteristics include controllable product morphology (controlled by adding a substrate), good crystallinity, and relatively uniform crystal sizes. These methods have broad applicability, overcoming the limitation of exfoliation methods, which can only be applied to layered materials. Traditional hydrothermal methods require high-temperature and high-pressure reactions that take a long time, consume significant energy, and pose safety risks. In contrast, microwave-assisted hydrothermal methods can achieve more precise heating, significantly improving production efficiency. Besides temperature, pressure, and reaction time, factors such as the type of sulfur source and metal precursor, and the hydrophilicity or hydrophobicity of the substrate surface ligands, all influence the outcomes of hydrothermal and solvothermal methods.

2.2.2. Chemical Vaporous Deposition (CVD)

Different from the hydrothermal and solvothermal methods based on liquid-phase reaction, chemical vapor deposition (CVD) is based on gas-phase reaction: gaseous elements and compounds containing thin-film elements decompose on the surface with catalysts, and further recombine to form thin films. This process often requires high temperature and pressure.

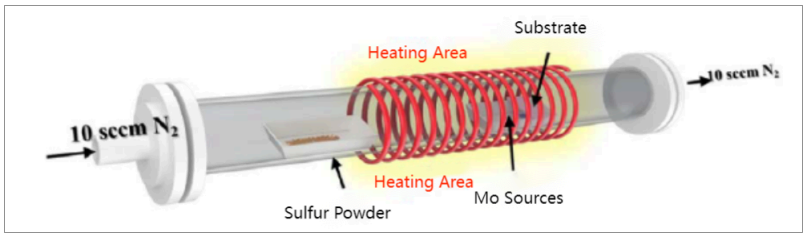


Figure 3: Experimental setup for the preparation of Mo S_2 by chemical vapor deposition [11]

Song used CVD to prepare large-area single-layer vertical Mo S_2 materials based on Si O_2 /Si, CC, and NF. The experimental setup is shown in Figure 3. The molybdenum source used was Mo O_3 . After pre-treating the substrate, CC requires oxidation and acid treatment, while others only need cleaning and drying) and placing the raw materials, N_2 air was purged for about 30 minutes. The flow rate of N_2 was adjusted to a constant of 10 sccm, and the temperature was increased at a rate of 5℃/min to 750℃, maintaining it for 30 minutes (NF only needs to be held for 5 minutes). The reaction was then cooled to room temperature, with the pressure in the tube maintained at approximately 35Pa throughout the process. Afterwards, the group grew nano-silver on Mo S_2 by reducing silver-ammonium solution and composited it with flexible substrates. Their production can be applied to detect various organic pollutants in gases and liquids [11].

Chemical vapor deposition is not limited to nanotransfer micromaterials, so its development has been relatively early and the technology is quite upgraded. The greatest advantage of this method is its ability to deposit single- or few-layer flat nanomaterials uniformly over quite large areas, with high production efficiency, so it's suitable for industrial-scale production. The main drawback lies in the need for a vacuum (or low-pressure) environment and high-purity gases, and the reaction temperature is typically around 800℃, thus limiting the choice of substrates.

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2.2.3. Electrochemical deposition

Most transition metal sulfides are ionic compounds, so the principle of electrochemical deposition can be referred to. The metal precursor, sulfur source (which can be hydrolyzed into S^{2-}) and reagents controlling solution pH can be dissolved together in deionized water to form an electrolyte, and then the nano-TMS material can be deposited by electrolysis.

Feng et al. prepared FeS micro/nano flowers using the electrochemical deposition method. Thiourea (providing a sulfur source, controlling pH and inhibiting oxidation of Fe^{2+}), ammonium ferric sulfate, and ascorbic acid (inhibiting oxidation of Fe^{2+}) were dissolved in deionized water under magnetic stirring for 20mins until fully dissolved. Electrochemical deposition was performed using a three-electrode system. The FeS nanomaterials produced by this method exhibit excellent gas-sensing performance [12].

Electrochemical deposition can be performed at room temperature, which is one of the greatest advantages over hydrothermal methods and CVD. What's more, by controlling the deposition time and voltage, the thickness of the deposit can be controlled precisely. However, its application range is relatively small because it's not suitable for forming covalent sulfides from less reactive metals. Edge effects also lead to uneven deposition.

In addition to the common deposition-based methods, there is another relatively special method for preparing quantum dots: thermal decomposition. Thermal decomposition involves forming nuclei and growing through the decomposition of reactants at higher temperatures in high-boiling-point solvents. When preparing TMS quantum dots, it's sulfur-containing transition metal complexes that are most commonly used as basic materials, which can yield nano TMS quantum dots upon heating. This method was discovered earliest and is one of the most effective preparation methods for quantum dots, but it may make subsequent processing more complex.

2.3. Biomineralization —an advanced preparation method

The biosynthesis of nano-TMS is also known as biomineralization. The core reaction involves biological metabolism rather than artificial chemical reactions, and it is catalyzed by enzymes, thus achieving high reaction efficiency. For the synthesis of nano-TMS, the most important microorganism is sulfate-reducing bacteria (SRB). These bacteria can use organic carbon sources, hydrogen, Fe^{2+} and other substances as electron donors to reduce sulfate ions in the environment to sulfite ions, which are then further reduced to S^{2-}. If there are also large amounts of transition metal ions in the reaction environment and an electric field guides their migration, they will combine further with $S^{2-}$and thus produce TMS. This method was first introduced for wastewater treatment [13], and later applied to the synthesis process of nano-TMS.

Zhao et al. used Postgate C medium to cultivate SRB bacteria for 8 days in an anaerobic, 30 ℃ environment. The copper mesh was ultrasonically cleaned in anhydrous ethanol and then soaked and assembled into an electrolytic cell. Then it was transferred to HCl to further remove oxides and then rinsed with large amounts of deionized water. Using the culture broth of SRB as the electrolyte, Pt as the counter electrode, saturated calomel electrode (SCE) as the reference electrode, and the copper mesh as the working electrode, the material was stirred and electrolyzed at a speed of 100rpm under anaerobic conditions to obtain flake-like Cu_2S nanomaterials. This material can efficiently degrade pollutants such as methylene blue and kill some harmful bacteria [14].

Biomineralization is one of the newest methods for preparing nano TMS. This approach only requires SRBs, a suitable environment to cultivate bacteria, and a moderate voltage to control ion migration. No high temperature, high pressure, or large current for electrolysis, so it is one of the most energy-efficient methods. What's more, biomineralization can be integrated with wastewater treatment,

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making it a win-win deal that we can process useful materials and deal with wastewater meanwhile. However, due to the variability of microbial communities and the rough understanding of how SRBs function, factors such as heavy metal ions in the environment may affect the performance of biomineralization. These factors result in a need for quality improvement in biomineralization synthesis.

2.4. Summary of different synthesis methods and selection of adaptation environment

It can be concluded from the above methods and their corresponding cases that there is no necessary superiority or inferiority among various synthesis methods. Different synthesis methods should be adopted for different application environments. The specific analysis can be started from the form of the product and combined with its function analysis.

① For porous or complex-shaped nano-TMS materials (such as electrodes, and specific structured catalysts), priority can be given to hydrothermal and solvothermal methods that produce uniformly crystallized structures, ensuring the crystals closely match the template. Electrochemical deposition can also be used, but this method may result in edge effects leading to uneven deposition. If the substrate has poor thermal resistance, high-temperature heating methods should not be employed.

② For planar thin-film nanotransistors, if the plane is large, chemical vapor deposition is preferred as it can generate crystalline films over a wide area with high efficiency; if the plane is small or used only for experiments rather than mass production, top-down epitaxy can be adopted, which is the simplest method; if precise control of the plane thickness is required (such as in various optoelectronic devices), electrochemical deposition is the preferred choice.

③ For the synthesis of quantum dots, hydrothermal and solvothermal methods and the thermal decomposition method are all good methods, among which the thermal decomposition method is more mature, while the hydrothermal and solvothermal methods consume less energy and are convenient for subsequent treatment.

④ For biofunctional materials, hydrothermal and solvothermal methods or biosynthesis methods can be selected, in which sodium citrate is also used to modify the surface of the material.

3. Problems existing in the preparation of nano-TMS

Although nano-TMS has shown promising application prospects in photoelectrics, catalysis, environmental protection and medicine, the scientific community's understanding of nanomaterials is not yet complete, and there are still loopholes in the synthesis and characterization at the nanoscale, so there are still deficiencies in the preparation of nano-TMS materials.

The primary challenge in the production of nano-TMS lies in controlling the manufacturing process: since at least one dimension of nanomaterials is on the nanoscale, even atomic-scale fluctuations can cause drastic changes in their physical and chemical properties. Any of these methods approach it from a macro perspective, making it difficult to maintain uniformity in size, morphology, and surface chemistry. For example, the thickness of Mo S_2 significantly affects its catalytic activity, the wavelength of light absorption, and the efficiency of converting electrical energy; Fe S_2 porous bodies synthesized using solvothermal synthesis tend to have easily passivated edges, leading to reduced cycling performance.

In addition to the production process, we’re also faced with the lack of testing methods. Due to the absence of in-situ characterization techniques and lack of recognized standards for the purity and dispersibility of nano-TMS, we cannot timely monitor the production process and products, which results in an unclear understanding of its growth mechanism, making it difficult to propose effective strategies for process optimizations. On the other hand, due to the feature of nanoparticles that they’re easy to aggregate and oxidize, whether the synthesized nano TMS materials can be used long-term and

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how they should be stored remain under question, and it further impacts the widespread adoption of nano-TMS.

In addition, controlling synthesis costs is also a significant challenge in the synthesis of nano-TMS. High temperatures and pressure are generally required during the synthesis process. High-purity gases are needed as protective gases and precision equipment is essential in chemical vapor deposition. Some precious metal sulfides (such as PtS_2) require expensive metals and substantial amounts of supports like substrates and solvents, thus making the synthesis cost disadvantageous.

Due to the transition elements being mostly heavy metals, including highly toxic elements such as Cd and Hg, these substances generally have poor biodegradability. Moreover, nanosheets and quantum dots exhibit strong diffusion properties, while metal precursors and sulfur-containing organic compounds also carry certain toxicity [15]. This leads to potential hazards for humans and the environment during the production of nano-TMS. How to avoid the toxicity of nano-TMS to personnel during production, while ensuring that waste liquids, gases, and solid waste left behind are treated harmlessly, may be one of the key breakthrough directions in current nano-TMS production.

In summary, the core issue that currently constrains the mass production of nano-TMS is how to balance the precision of production, the efficiency of mass production, the cost of production and the potential impact on health and the environment.

4. Conclusion

This article summarizes common methods for the preparation of nano-TMS, including the more traditional top-down method (exfoliation), the commonly used bottom-up method in precision preparation, and the newer and environmentally friendly biomineralization synthesis. It also addresses the reasons why nano-TMS is currently limited to experimental and small-scale preparation, making it difficult to scale up for large-scale production. To promote the large-scale production and application of nano-TMS, the most important step is to change the mindset—by summarizing the shortcomings and issues in various production methods, shifting from "empirical trial and error" to "theoretical design." The following is a possible outlook on the transition of nano-TMS to large-scale production:

① The reaction space is highly quantified and miniaturized, dividing larger reaction systems into multiple smaller microreactors. By leveraging microfluidic technology [16], real-time monitoring of changes in reaction conditions is achieved. Additionally, atomic layer deposition (ALD) techniques, which are used for manufacturing high-precision equipment, are introduced into the preparation process of nano-TMS materials, enabling atomic-level monitoring and control.

② Increase research on biomineralization to fully understand the mechanisms and structures of related substances involved. Combine genetic engineering and other technologies to enhance the production efficiency and environmental adaptability of relevant strains, or achieve artificial control reactions through the synthesis of similar enzymes. At the same time, in the recovery of by-products such as waste liquids, use solar energy decomposition and ion adsorption to convert them back into raw materials for further production, achieving a closed-loop and green cycle throughout the process.

③ With the help of artificial intelligence technology, AI will gradually participate in reaction condition control and product characterization, so as to realize mass production and automation.

The synthesis of nano-TMS is an interdisciplinary science that integrates materials science, chemical engineering, environmental and biological engineering, as well as big data. We need to summarize various experimental experiences while integrating relevant technologies from multiple fields, to provide a comprehensive theoretical foundation and synthesis standards for the synthesis of nano-TMS, and further guide it from the laboratory to our daily lives.

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