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Synthesis of WO_3 Thin Films by Spray Pyrolysis Integrated with Artificial Intelligence and Machine Learning for Process Optimization

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Abstract

Tungsten trioxide (WO_3) is an important transition metal oxide widely used in gas sensors, electrochromic smart windows, photocatalysis, and optoelectronic devices due to its tunable band gap, high chemical stability, and excellent surface reactivity. Spray pyrolysis is a cost-effective and scalable technique for depositing WO_3 thin films; however, achieving optimal structural and functional properties strongly depends on multiple interdependent deposition parameters. Recently, Artificial Intelligence (AI) and Machine Learning (ML) techniques have emerged as powerful tools for materials optimization and predictive synthesis.This work presents a comprehensive study on the fabrication of WO_3 thin films using spray pyrolysis combined with AI/ML-assisted optimization. Experimental parameters such as precursor concentration, substrate temperature, spray rate, carrier gas pressure, and annealing conditions are modelled using supervised learning algorithms to predict film morphology, crystallinity, optical band gap, and sensing performance. Machine learning models including Artificial Neural Networks (ANN), Support Vector Regression (SVR), and Random Forest algorithms enable predictive optimization and adaptive synthesis control. The proposed AI-guided framework significantly reduces experimental trials while improving film quality and device performance. The integration of spray pyrolysis with intelligent data-driven optimization represents a promising pathway toward autonomous thin-film fabrication systems.

Keywords: WO_3 thin films, Spray pyrolysis, Artificial intelligence, Machine learning, Smart materials.

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Introduction

Tungsten trioxide (WO_3) is an n-type semiconductor that has attracted considerable attention due to its versatile electrochromic, sensing, catalytic, and photoelectrochemical properties. It finds applications in gas sensors, smart windows, solar energy conversion, photocatalysis, and optoelectronic devices. In particular, thin-film WO_3has become a focus of research because its electrical and optical behavior is highly dependent on deposition conditions and microstructural features. Among the various deposition techniques, spray pyrolysis stands out for its advantages, including low fabrication cost, suitability for large-area coatings, industrial scalability, and the ability to control composition.

Despite these benefits, the properties of WO_3 films remain extremely sensitive to synthesis parameters such as substrate temperature, precursor molarity, spray rate, and annealing temperature. Even minor variations in these factors can significantly influence crystallinity, grain size, and band gap energy. Traditionally, optimizing these parameters through trial-and-error methods has been labour-intensive and time-consuming.

In recent years, Artificial Intelligence (AI) and Machine Learning (ML) have emerged as powerful data-driven approaches capable of predicting material properties and automatically guiding synthesis conditions. This paradigm of AI-guided materials synthesis is increasingly recognized as a cornerstone of intelligent manufacturing and autonomous laboratories, offering a pathway toward faster, more efficient, and more precise development of advanced functional materials [1-3].

2. Fundamentals of WO_3 Thin Films

2.1 Material Properties of WO_3

WO_3 thin films possess distinctive material properties that make them highly attractive for functional applications. The band gap of WO_3 typically lies in the range of 2.6–3.0 eV, enabling effective interaction with visible light and supporting its use in electrochromic and photoelectrochemical devices. Structurally, WO_3 can exist in multiple crystalline phases, including monoclinic, tetragonal, and hexagonal forms, each imparting different physical characteristic. A key feature of WO_3 is its strong sensitivity to oxygen vacancies, which play a critical role in determining electrical conductivity, optical absorption, and catalytic activity. The electrochromic response of WO_3 is particularly noteworthy, as it allows reversible changes in optical properties under applied voltage, making it ideal for smart window technologies[4-5].

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In thin-film form, the physical properties of WO_3 are strongly influenced by deposition conditions, especially the control of oxygen vacancies and grain morphology. Films synthesized via spray pyrolysis generally exhibit polycrystalline monoclinic structures after annealing, and this structural arrangement directly impacts both optical transparency and electrical performance. Consequently, understanding and tailoring the interplay between crystalline phase, grain size, and defect concentration is essential for optimizing WO_3 thin films for advanced optoelectronic and energy-related applications.[6]

3. Spray Pyrolysis Technique

3.1 Principle

The spray pyrolysis technique is a versatile and widely used method for fabricating WO_3 thin films. Its principle is based on the atomization of a precursor solution into fine droplets, which are then directed onto a heated substrate. Upon contact, these droplets undergo thermal decomposition, leading to the formation of WO_3 along with gaseous byproducts. The overall reaction can be represented as:

Wprecursor → WO_3 + gaseous byproducts

A variety of tungsten-based precursors can be employed in this process, with common examples including tungsten hexachloride (WCl_6), ammonium metatungstate, and peroxotungstic acid. The choice of precursor, along with deposition parameters such as substrate temperature and spray rate, plays a crucial role in determining the structural, optical, and electrical properties of the resulting thin films. Spray pyrolysis thus offers a simple yet effective route for producing WO_3 coatings, combining low fabrication cost with scalability and compositional control, making it highly suitable for both research and industrial applications.[7-8]

3.2 Deposition Parameters

The important synthesis variables for WO_3 thin films and their optimized influence on film properties:

Parameter	Influence on Film	Optimized Condition
Substrate temperature	Crystallinity	350–450 °C (ensures well-crystallized films)
Spray rate	Thickness	Moderate rate (~3–5 mL/min for uniform films)
Solution molarity	Grain size	0.05–0.1 M (controls fine grain growth)

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Parameter	Influence on Film	Optimized Condition
Carrier gas pressure	Uniformity	Stable, moderate pressure (~1 atm)
Annealing temperature	Phase formation	400–500°C (promotes monoclinic phase

Table1: synthesis variables for WO_3 thin films and their optimized influence on film properties

Optimizing these parameters is crucial for tailoring the electrochromic and sensing performance of WO_3 thin films. Proper control ensures desirable crystallinity, uniform thickness, fine grain morphology, and stable phase formation, all of which directly enhance optical transparency, conductivity, and device efficiency.[9-10]

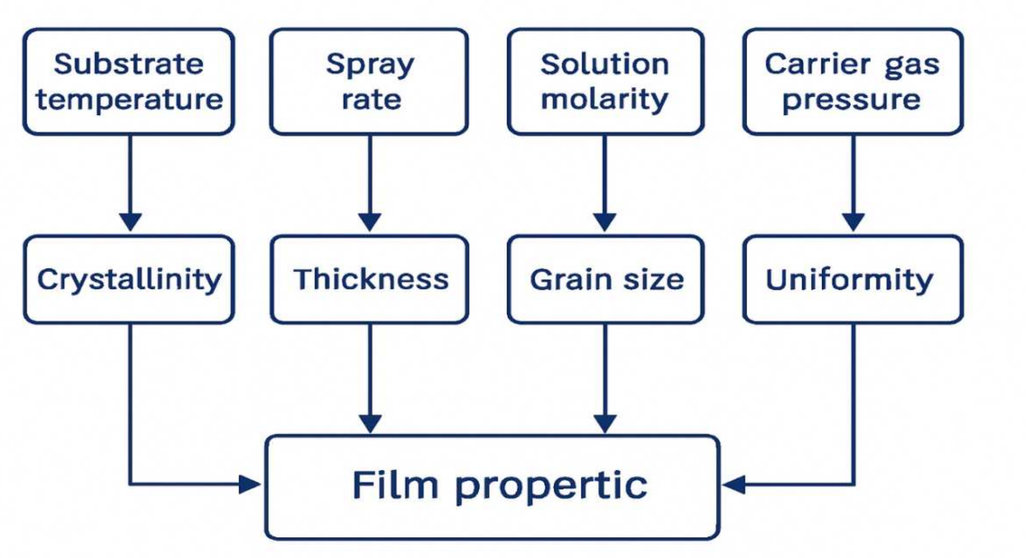

Figure1: synthesis parameter in spray pyrolysis directly influences WO_3 thin-film properties [11]

3.3 Structural Characteristics

concise explanation for each structural characteristic of spray-deposited WO_3 thin films:

- **Nanogranular morphology**: The films consist of nanoscale grains, which increase surface area and enhance interactions with light and gases—beneficial for sensing and photocatalytic applications.

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- **Polycrystalline structure**: WO_3 films typically form multiple crystalline domains, often in the monoclinic phase, contributing to mechanical stability and tunable electronic properties.
- **Tunable optical transparency**: The transparency of the films can be adjusted by controlling grain size, thickness, and oxygen vacancies, making them ideal for electrochromic devices like smart windows.
- **Variable electrical conductivity**: Conductivity depends on factors such as crystallinity, defect density, and doping, allowing customization for different electronic and sensing applications.
- **Effect of doping and precursor chemistry**: Introducing dopants or altering precursor composition can significantly modify the optical band gap and crystallite size, enabling precise control over film performance.[12]

Dopant	**Effect on Band Gap**	**Effect on Crystallite Size / Morphology**	**Effecton Conductivity / Electrochromic Performance**	**Notes**
Ti	Slightly increases band gap (toward ~3.0 eV)	Leads to smaller crystallite size, refined nanostructure	Enhances electrochromic contrast and cycling stability	Ti-doping improves transparency and durability of WO_3 films
Mo	Narrows band gap (toward ~2.6 eV)	Promotes larger grains and improved crystallinity	Increases electrical conductivity, beneficial for sensing	Mo-doping enhances charge transport and reduces coloration time
Nb	Slightly reduces band gap	Increases oxygen vacancy concentration, modifies grain boundaries	Improves ionic/electronic conductivity, boosting electrochromic speed	Nb-doping is often used to accelerate switching response in smart windows

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Table 2: **Different dopants (Ti, Mo, Nb) influence the structural and functional properties of WO_3 thin films[13]**

- **Ti-doping** is best for improving optical transparency and long-term stability.
- **Mo-doping** enhances conductivity and reduces band gap, making films more suitable for sensing and photocatalysis.
- **Nb-doping** increases vacancy concentration and conductivity, which accelerates electrochromic switching.[140

4. Artificial Intelligence and Machine Learning in Thin Film Synthesis

4.1 Need for AI Integration

Traditional optimization of WO_3 thin-film synthesis is often challenging because the material properties depend on multiple nonlinear variables such as substrate temperature (T), solution concentration (C), spray rate (R), carrier gas pressure (P), and annealing time (t). In practice, this means that hundreds of experiments are usually required to identify the right combination of parameters, making the process time-consuming and resource-intensive. Artificial Intelligence (AI) and Machine Learning (ML) offer a transformative solution to this problem. Instead of relying solely on trial-and-error, AI models can directly learn the complex relationships between synthesis parameters and film properties from experimental datasets. By capturing these nonlinear dependencies, AI enables accurate predictions of outcomes such as crystallinity, grain size, band gap, and conductivity. This data-driven approach not only accelerates optimization but also guides researchers toward intelligent synthesis strategies, marking a significant step toward autonomous laboratories and smart manufacturing in thin-film materials science.[15-17]

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4.2 Machine Learning Workflow

AI-guided WO_3 synthesis framework

Figure2: Stepwise flow highlights how experimental data and machine learning interact to continuously refine synthesis conditions [18]

5. Machine Learning Models for WO_3 Optimization

5.1 Artificial Neural Network (ANN)

Artificial Neural Networks (ANNs) play a pivotal role in optimizing WO_3 thin-film synthesis by modeling complex, nonlinear relationships between deposition parameters and material properties. These models are particularly effective in predicting key outputs such as band gap, grain size, electrical conductivity, and sensor response, which are all influenced by variables like substrate temperature, precursor concentration, and annealing conditions.The strength of ANN lies in its ability to minimize prediction error by learning from experimental datasets. This is achieved through an error function defined as:

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$$E = \sum(y_{exp} - y_{pred})^2$$

Where,

(y_{exp}) represents the experimentally measured values

(y_{pred}) denotes the model's predictions.

By iteratively adjusting internal weights and biases, the ANN reduces this error, resulting in increasingly accurate predictions. This capability makes ANN a powerful tool for guiding synthesis decisions, reducing experimental workload, and accelerating the development of high-performance WO_3 thin films.[19-20]

5.2 Support Vector Regression (SVR)

Effective for small experimental datasets:

- High generalization ability
- Accurate property prediction

Support Vector Regression (SVR) is a powerful machine learning technique particularly well-suited for WO_3 thin-film optimization when working with small experimental datasets. Unlike models that require extensive data to achieve accuracy, SVR excels in scenarios where data is limited, thanks to its high generalization ability. This means it can effectively capture the underlying trends and relationships between synthesis parameters and material properties without overfitting.SVR is capable of delivering accurate predictions for key outputs such as band gap, grain size, and conductivity, even when the dataset is sparse. Its robustness and precision make it a valuable tool in early-stage research, where experimental data is costly or time-consuming to obtain. By integrating SVR into the optimization workflow, researchers can accelerate material discovery and reduce the number of required experiments while maintaining predictive reliability.[21-22]

5.3 Random Forest Algorithm

Useful for parameter importance analysis:

Example ranking:

1. Substrate temperature
2. Annealing temperature
3. Precursor concentration

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The Random Forest algorithm is a valuable machine learning tool for optimizing WO_3 thin-film synthesis, particularly when the goal is to understand the relative importance of different deposition parameters. By constructing an ensemble of decision trees, Random Forests can evaluate how strongly each parameter influences material properties such as band gap, grain size, and conductivity. This makes them especially useful for parameter importance analysis, where the algorithm ranks variables based on their contribution to predictive accuracy. For example, Random Forest analysis often highlights substrate temperature as the most critical factor, followed by annealing temperature and precursor concentration. These rankings provide researchers with clear guidance on which synthesis variables should be prioritized during optimization. By focusing on the most influential parameters, experimental efforts can be streamlined, reducing trial-and-error and accelerating the development of high-performance WO_3 films.[23-25]

6. AI-Assisted Spray Pyrolysis Optimization

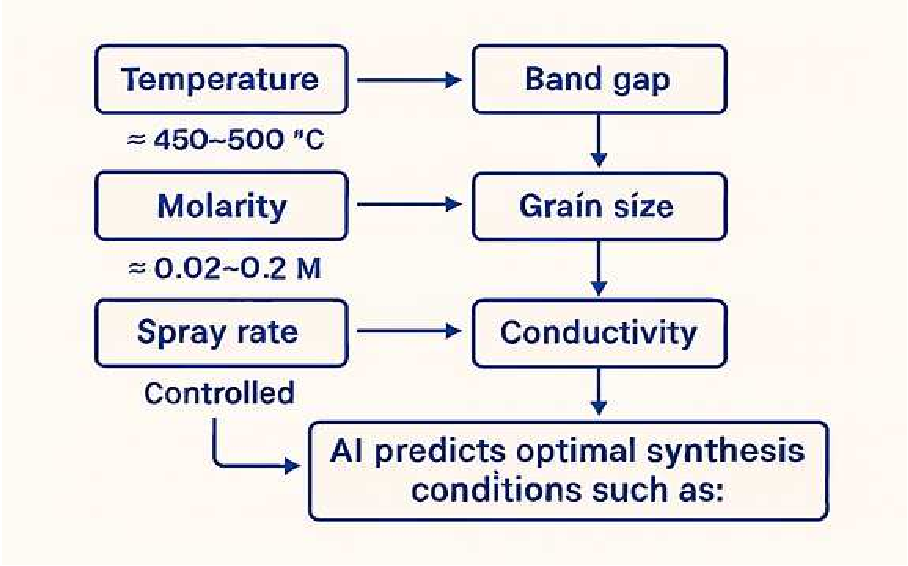

Figure 3: AI predicts optimal synthesis conditions [26]

7. Applications of AI-Optimized WO_3 Films

- **Gas Sensors**: Ag-doped WO_3 films → high NH_3 sensitivity, fast response (~12 s).
- **Smart Windows**: Electrochromic WO_3 films → stable optical modulation, durable over cycling.

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- **Photocatalysis**: Nanostructured WO_3 → improved surface reactions, enhanced efficiency. 27-28]

8. Proposed Intelligent Autonomous Deposition System

- Real-**time sensors** → continuous monitoring of film growth conditions.
- Closed-**loop ML optimization** → machine learning adjusts parameters based on feedback.
- **Automated parameter adjustment** → deposition variables (temperature, spray rate, dopant level) tuned instantly.
- **Self-learning fabrication** → system improves with each cycle, refining film quality.
- **Outcome** → moves toward **self-driving materials laboratories**, enabling autonomous discovery and synthesis.[29-30]

Results and Expected Advantages

Integrating AI into thin-film deposition significantly improves research efficiency and outcomes. It reduces experimental costs by minimizing waste and repetitive trials, while machine learning accelerates optimization of deposition parameters. Automated control enhances reproducibility, ensuring consistent film quality across cycles. At the same time, AI-guided fabrication delivers superior thin-film properties, including uniformity and performance. Beyond immediate improvements, AI enables predictive material discovery, allowing researchers to identify promising new compositions and structures more quickly. Together, these advantages drive the field toward autonomous, self-driving materials laboratories.[31-33]

10. Future Research Directions

Future work in AI-driven thin-film deposition aims to push toward fully autonomous fabrication. Reinforcement learning can enable deposition systems that adapt and improve through continuous feedback. Digital twins will provide virtual models of thin-film growth, allowing predictive simulations and real-time control. Edge-AI controlled spray systems will bring intelligence directly to the deposition hardware for faster, localized decision-making. Ultimately, these advances converge toward autonomous nanomaterial fabrication, where AI platforms independently design, synthesize, and optimize materials for next-generation applications.[34-35]

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11. Conclusion

WO_3 thin films synthesized by spray pyrolysis provide a versatile foundation for functional devices across sensing, energy, and optoelectronic applications. Traditional optimization methods are limited by the complex interdependence of synthesis parameters, but the integration of Artificial Intelligence and Machine Learning offers powerful predictive and adaptive control. By enabling intelligent deposition processes, AI transforms spray pyrolysis into a next-generation manufacturing approach, accelerating material discovery, enhancing film quality, and improving overall device performance. This convergence of AI and thin-film synthesis marks a significant step toward smart, autonomous materials laboratories.[36-37]

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