Fabrication and Gas Sensing Studies of Metal Oxide-Based Sensors with LabVIEW Automation

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CERTIFICATE

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Project guide

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ABSTRACT

The project focuses on the fabrication and gas sensing studies of metal oxide-based sensors, with a specific focus on employing LabVIEW for automation. Metal oxide semiconductor gas sensors were fabricated and assessed for their gas sensing capabilities, using materials such as TiO_2 and ZnO. These sensors were then tested in gas sensing experiments to assess their efficacy in detecting gases like ammonia. Furthermore, LabVIEW-based software was developed to sensing procedure, facilitating the automate streamlined data acquisition and analysis. The abstract underscores the significance of metal oxide semiconductor gas sensors across various domains, including environmental monitoring, industrial safety, and healthcare. By integrating LabVIEW automation, the project aims to optimize the efficiency and dependability of gas sensing processes, rendering them suitable for real-time monitoring and control applications.

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CHAPTER 1 INTRODUCTION

1.1 INTRODUCTION

A sensor is an item which picks up a stimulus and reacts to it by producing electricity. The output signals represent some forms of electrical signal, such as current or voltage. A detector is a device that accepts many types of signals like physical, chemical or biological and changes them into electric signals.

The kinds of sensors are categorized in accordance with their applications, input signal, conversion mechanism, material used for sensor properties (costs range accuracy) among others like this chapter does give information on different classifications based on; thermal magnetic optical mechanical and chemical detectors.

Sensors come in two main types: passive and active. Passive sensors don't need any extra power source - they generate an electrical signal directly in response to external stimuli. This means they convert input energy into output signal energy. Examples of passive sensors include photographic, thermal, electric field, chemical, infrared, and seismic sensors. Active sensors, on the other hand, require an external energy source to respond, known as an excitation signal. They produce output signals by making necessary changes to these input signals. There are also other sensor types based on their detection properties, such as variation mechanism (e.g., photoelectric, thermoelectric, electrochemical, electromagnetic), and whether they are analog or digital. Sensors can detect various physical, chemical, electric, and magnetic phenomena [1].

Analog sensors produce an analog output, i.e. continuous output signals are produced with respect to the measured quantity, but a digital sensor is the opposite of analog sensors, with discrete characteristics and digital output in nature. As said before there are many sensors that are specific made to sense physical quantities and objects.

Thermal sensors sense the flow of heat, position sensor can sense the position of an object, accelerometer can sense acceleration, touch sensors are sensitive to touch, pressure, etc. [3].

Chemical Sensor: A chemical sensor is a device that detects and reports chemical information. This information can be about the composition, concentration, or activity of a chemical. Chemical sensors have many applications, such as in home appliances and the chemical industry.

Chemical sensors have two main components: a chemical receptor and a physicalchemical transducer. The receptor interacts with the target molecules, while the transducer converts this interaction into an electrical signal.

Here's how a chemical sensor works: A test sample is given to the receptor, which analyzes its composition. The transducer then collects this information from the receptor and sends it to a signal amplifier. The amplifier strengthens the signal from the transducer and outputs it as the final reading [1, 2].

Gas sensor: Gas sensors play a crucial role in various industries and applications where the detection and monitoring of gases are required. These sensors provide valuable information about the presence and concentration of different gases in the surrounding environment (Shi et al., 2020). Legislation has led to an increased demand for gas sensors in environmental monitoring, particularly in the workplace and areas affected by industrial effluents or agricultural runoff. The function of a gas sensor is to analyze the physical, chemical, and biological environment by providing information about the presence and concentration of specific gases.

Gas sensors consist of two main components: a physical transducer and a chemically selective layer. The physical transducer in a gas sensor is responsible for converting the gas analyzed into a measurable signal, while the chemically selective layer is designed to interact with specific gas molecules. This interaction results in a change that is detected by the transducer, thus providing a quantifiable output.

Gas sensors are crucial devices used in various industries like environmental monitoring, safety, automotive, and healthcare. They can detect many different gases such as carbon monoxide, methane, ammonia, and volatile organic compounds. As technology advances, developing more sensitive, selective, and reliable gas sensors is essential for ensuring safety and efficiency across different sectors. These sensors convert gas concentration or other gas-related parameters into an electrical signal that can be measured and analyzed [6].

Among the various gas sensor types, thin-film semiconductor coated sensors have become a promising technology. They are known for their sensitivity, stability, and ability to detect specific gases. This text explores the working principle, examples, and applications of these thin-film semiconductor coated gas sensors, as well as their broader usage in industrial, medical, and scientific domains.

Gas sensors made from thin semiconductor coatings, known as metal oxide gas sensors, work by detecting gas molecules that stick to the semiconductor surface, changing its electrical conductivity. These sensors typically have a semiconductor material covered in a thin metal oxide layer, like tin dioxide or tungsten oxide. When exposed to a target gas, the gas molecules adhere to the semiconductor surface, altering the film's conductivity. This conductivity change is then measured and linked to the concentration of the target gas [1, 7].

One example of a thin film semiconductor coated gas sensor is the SnO_2 -based sensor used for detecting carbon monoxide (CO). When CO molecules adsorb onto the SnO_2 surface, they capture free electrons, leading to a decrease in the sensor's conductivity. This change is detected and translated into a measurable signal, indicating the presence of CO gas.

Another example is the WO_3 -based sensor used for detecting nitrogen dioxide (NO_2). Similar to SnO_2 , WO_3 undergoes a change in conductivity when exposed to NO_2 , allowing for the accurate detection of this harmful gas in the environment.

1.2 Applications of gas sensor

Thin film semiconductor coated gas sensors find applications across various industries and sectors:

- Environmental Monitoring: These sensors are used for monitoring air quality in urban areas, detecting pollutants such as CO, NO₂, and volatile organic compounds (VOCs).
- Industrial Safety: Gas sensors are employed in industries such as manufacturing, petrochemicals, and mining to detect leaks of toxic or flammable gases, ensuring worker safety and preventing accidents.
- Automotive: Gas sensors are integrated into vehicle exhaust systems to monitor emissions and ensure compliance with environmental regulations.
- Medical: Gas sensors are utilized in medical devices for monitoring patient health, detecting gases associated with respiratory conditions, and controlling anesthesia levels during surgeries.
- Food Industry: Gas sensors help in monitoring food quality by detecting spoilage gases produced during storage and transportation.

In gas detection technology, sensors with gas-sensitive metal oxides as their transducing layer are a remarkable landmark since they feature high sensitivity to a variety of gases and chemicals. They have been used in different areas like car exhaust monitoring systems for environmental reasons, accelerometers in vehicle airbag systems to improve safety during accident processes as well as in breath analysis equipment for medical diagnosis purposes. As the need for reliable gas detection grows, these sensors will play an increasingly vital role in ensuring a safer, healthier world for all.

Chemical sensitivity: Through it's various applications we know that Gas sensors play a pivotal role in various industries and everyday life, ensuring safety, environmental monitoring, and industrial processes. And for which the most important property required is their Chemisensitivity. Among them, thin film semiconductor coated gas sensors stand out for their exceptional chemisensitivity.

Chemical sensitivity refers to the sensor's ability to detect and differentiate between different gases or vapors. Understanding and improving the chemical sensitivity of gas sensors is crucial for enhancing their accuracy and reliability in various applications.

There are several factors that contribute to the chemical sensitivity of gas sensors, including the type of sensing material, the surface area of the sensing material, and the interaction between the gas molecules and the sensing material. Research in this field aims to develop new sensing materials with tailored properties to improve the selectivity and sensitivity of gas sensors. Additionally, advances in nanotechnology have opened up new possibilities for enhancing the chemical sensitivity of gas sensors by manipulating the structure and composition of sensing materials at the nanoscale [7].

Furthermore, the integration of gas sensors with advanced signal processing techniques and data analysis algorithms has the potential to improve their chemical sensitivity and overall performance. By combining the strengths of material science, nanotechnology, and data analysis, researchers are continually pushing the boundaries of gas sensor technology to meet the increasing demands for more reliable and precise gas detection systems.

Detection Principle: Thin film semiconductor-coated gas sensors work by detecting changes in electrical conductivity when exposed to target gases. The semiconductor thin film acts as the sensing material, with its conductivity varying depending on the presence of different gases. This change occurs because gas molecules are either absorbed or released from the thin film surface, altering its electrical properties. When the target gas interacts with the semiconductor surface, it either captures or releases electrons, leading to a change in conductivity. This change is then measured and correlated to the gas concentration.

Metal oxide gas sensors, particularly those exhibiting semiconducting properties, currently stand at the forefront of gas sensor research. This category garners substantial attention in atmospheric gas sensing due to several key factors: their cost-effectiveness, ease of production, simplicity in operation, and versatility in detecting a wide range of gases across various application domains.

Moreover, beyond the conventional conductivity-based detection mechanism, gassolid interactions can be discerned through alterations in capacitance, work function, mass, optical properties, or the energy released during the reaction. These diverse detection methods provide a comprehensive approach to gas sensing, catering to different sensor requirements and environmental conditions.

The interpretation of measured values typically involves electrode systems, diode configurations, transistor setups, surface wave components, thickness-mode transducers, or optical setups. This variety in read-out methods ensures flexibility in sensor design and compatibility with different sensing principles.

Furthermore, Chemical processes that rely on specific reactions with a substance are commonly used to detect gases. These solid-state chemical detection methods play a crucial role in making gas sensing applications more targeted and sensitive, contributing to the advancement of sensor technology [7-8].

Many metal oxides can be used to detect flammable, reducing, or oxidizing gases by measuring changes in their electrical conductivity. Some examples of these metal oxides that show a response to gases include chromium oxide (Cr_2O_3) , manganese oxide (Mn_2O_3) , cobalt oxide (Co_3O_4) , nickel oxide (NiO), copper oxide (CuO), strontium oxide (SrO), indium oxide (In_2O_3) , tungsten oxide (WO_3) , titanium dioxide (TiO_2) , vanadium oxide (V_2O_3) , iron oxide (Fe_2O_3) , germanium oxide (GeO_2) , niobium oxide (Nb_2O_5) , molybdenum oxide (MoO_3) , tantalum oxide (Ta_2O_5) , lanthanum oxide (La_2O_3) , cerium oxide (CeO2), and neodymium oxide (Nd_2O_3) .

Metal oxides selected for gas sensors can be determined from their electronic structure. The range of electronic structures of oxides is so wide that metal oxides were divided into two the following categories:

Transition-metal oxides (Fe₂O₃, NiO, Cr₂O₃, etc.)

Non-transition-metal oxides, which include (a) pre-transition-metal oxides (Al_2O_3 , etc.) and (b) post-transition-metal oxides (ZnO, SnO₂, etc.) [9].

Transition-metal oxides behave differently because the energy difference between a cation dn configuration and either a dn+1 or dn-1 configurations is often rather small. Thus have greater sensitivity.

Selectivity:

Selectivity refers to a sensor's ability to distinguish between different gases. MOX gas sensors exhibit selectivity primarily due to their surface chemistry, which interacts differently with various gas molecules. The selectivity of MOX sensors can be enhanced through material design, surface functionalization, and signal processing techniques. For instance, researchers have developed nanostructured MOX materials with tailored surface properties to improve selectivity towards specific gases like methane, carbon monoxide, or volatile organic compounds (VOCs). Additionally, advanced signal processing algorithms, such as pattern recognition techniques, can enhance selectivity by analyzing the sensor response patterns to different gases. [6-7]

Sensitivity:

How easily a sensor can detect tiny amounts of a target gas is referred to as sensitivity in MOX gas sensors. Because of their large surface area and high surface to volume ratio enabling them to interact with gas molecules optimally, MOX gas sensors are usually very sensitive. They can become more sensitive through development of their constituency which entails adding other substances for improving their ability of gas adsorption as well as electron mobility while still improving it as they are adsorbed on them up to a certain extent by doing that. Moreover, advancements in nanotechnology have led to the development of nanostructured MOX materials with enhanced sensitivity and reduced response times. [6-7]

Response Time:

Response Time of MOX Gas Sensors Response time refers to the time taken by a sensor to detect and respond to changes in gas concentration. The response time of MOX gas sensors depends on various factors, including the diffusion of gas molecules to the sensor surface, adsorption kinetics, and the electrical response of the sensing

material. Typically, MOX sensors exhibit rapid response times, making them suitable for real-time monitoring applications. However, response time can vary depending on factors such as operating temperature, humidity, and the presence of interfering gases. Researchers have explored strategies to improve the response time of MOX sensors, such as optimizing operating conditions, modifying the sensor structure, and integrating temperature and humidity compensation techniques [5].

Conclusion In conclusion, understanding the selectivity, sensitivity, and response time of MOX gas sensors is essential for their effective deployment in various applications. Advances in material science, nanotechnology, and signal processing techniques continue to enhance the performance of MOX sensors, making them indispensable tools for gas detection and monitoring. Further research and development efforts are warranted to address challenges related to selectivity, sensitivity, and response time, ultimately advancing the capabilities of MOX gas sensing technology.

1.3 Metal Oxides

Zinc oxide:

Zinc oxide serves a critical role as an oxide semiconductor in detecting harmful and flammable gases. Sensor components utilizing ZnO come in diverse configurations including single crystals, pellets, thick films, and thin films. The sensing process hinges on the absorption of oxygen on the oxide's surface, followed by electron transfer when reacting with the gas molecules. This interaction alters the surface resistance of the sensor element, with thin ZnO films demonstrating heightened sensitivity in gas detection compared to other forms.

Zinc oxide stands out as a widely researched material for gas sensing due to its affordability, non-toxic nature, and accessibility through conventional chemical synthesis methods. Additionally, ZnO has the capability to create heterostructures,

enhancing sensor performance in terms of sensitivity, selectivity, and stability. Furthermore, these heterostructures aid in reducing the operating temperature of gas sensors, as the combined effects work synergistically to amplify the sensor signal. [9]

Titanium dioxide:

Titanium dioxide (TiO₂) is a widely used material in gas sensors. This is because of its excellent sensing properties, particularly in detecting gases like hydrogen (H₂), carbon monoxide (CO), and volatile organic compounds (VOCs). TiO2 has a large surface area, which provides plenty of active sites for gas molecules to attach to its surface.

When a reducing gas touches the TiO_2 surface, it sticks to it, leading to changes in TiO_2 's electronic properties. TiO_2 is an n-type semiconductor, meaning it has extra electrons. As the reducing gas molecules attach to the surface, they capture some of these electrons, reducing the electron concentration and decreasing the conductivity.

This change in conductivity is measured by passing a small electrical current through the TiO₂ film. As gas molecules adsorb, the resistance of the TiO₂ film increases, changing the measured current. By monitoring this change, the presence and amount of the target gas can be determined. The selectivity of TiO₂-based sensors can be improved by doping the TiO₂ with other metal oxides or by using composite materials. This allows for tailored responses to specific gases and reduces cross-sensitivity to other gases. The sensing properties of TiO₂-based sensors are often temperaturedependent. Typically, these sensors operate at elevated temperatures (around 200-500°C) to enhance gas adsorption and desorption kinetics, as well as to improve sensitivity and selectivity [4, 9].

Sensing Mechanism: The mechanism of gas sensing is a widely debated subject but it is essentially trapping electrons at adsorbed molecules and band bending induced by these charged molecules are responsible for a change in conductivity. The negative

charge trapped in these oxygen species causes an upward band bending and thus a reduced conductivity compared to the flat band situation. When O2 molecules are adsorbed on the surface of metal oxides, they would extract electrons from the conduction band Ec and trap the electrons at the surface in the form of ions. This will lead to a band bending and an electron-depleted region. The electron-depleted region is the so-called space-charge layer, of which thickness is the length of the band bending region. Reaction of these oxygen species with reducing gases or a competitive adsorption and replacement of the adsorbed oxygen by other molecules decreases and can reverse the band bending, resulting in an increased conductivity. O– is believed to be dominant at the operating temperature of 300–450 °C which is the work temperature for most metal oxide gas sensors. When gas sensors are exposed to the reference gas with CO, CO is oxidized by O– and released electrons to the bulk materials. Together with the decrease of the number of surface O–, the thickness of the space-charge layer decreases. Then it would be easy for electrons to conduct in sensing layers [10, 11].

1.4 Literature Review

Metal oxide semiconductor sensors have become a focus of research because of their many advantages. These sensors can detect changes in the electrical conductivity of metal oxide films when exposed to target gases. They offer high sensitivity, fast response, and compatibility with micro fabrication.

Among the various metal oxides studied, titanium dioxide (TiO_2) stands out. It has a large surface area, is chemically stable, and its electronic characteristics can be adjusted. Extensive research has been done on TiO_2 -based gas sensors. These sensors have shown high sensitivity and selectivity in detecting a wide range of gases, such as

volatile organic compounds (VOCs), nitrogen dioxide (NO₂), carbon monoxide (CO), and hydrogen (H₂). This indicates their potential for diverse applications, including environmental monitoring, industrial safety, and healthcare.

Titanium dioxide (TiO_2) gas sensors work by detecting changes in electrical conductivity when target gas molecules interact with the TiO_2 surface. These interactions are influenced by factors like surface defects, oxygen gaps, and the process of gases being absorbed and released.

Understanding these processes is crucial for optimizing sensor performance for specific gas detection needs. Recent advancements in TiO_2 gas sensors include developing nanomaterial, adding metal nanoparticles, and integrating them with microelectronic platforms. These improvements aim to enhance sensing capabilities, miniaturize devices, and address challenges like selectivity, stability, response time, and environmental interference.

However, challenges remain in the field of TiO_2 gas sensors. These include improving selectivity, stability, and response time, as well as addressing issues like sensor poisoning and environmental interference. Additionally, there is a need for standardizing sensor fabrication and testing protocols to ensure consistent and reproducible results across studies.

The study of TiO_2 -based gas sensors shows promise for many applications, including the detection of volatile liquids. By understanding how these sensors work and improving their performance, this research could help develop advanced sensing devices for real-world use in environmental monitoring, industrial safety, and healthcare.

1.5 Motivation

The need for sensitive and accurate sensors to detect volatile liquid materials is becoming increasingly pressing. Researchers are exploring new ways to develop improved sensor devices that can meet the demands of environmental conservation, industrial production, and healthcare. Metal oxide gas sensors, like those based on titanium dioxide (TiO₂), show great promise due to their ability to detect subtle changes in gas composition.

This research focuses on creating highly sensitive and selective sensors capable of detecting volatile liquid compounds. TiO_2 is a particularly promising sensing material, as it offers remarkable physical and chemical stability, large surface area, and the capacity to interact with gas molecules. By harnessing the untapped capabilities of TiO_2 , the researchers aim to develop innovative sensing solutions to address the pressing needs in environmental monitoring, industrial processes, and public health surveillance.

Titanium dioxide (TiO_2) is a promising material for developing new sensing technologies. It is abundant, inexpensive, and easy to manufacture. This study aims to unlock the sensing capabilities of TiO_2 for detecting volatile liquid substances by combining scientific research, empirical testing, and technological advancements. This could lead to a significant shift in gas sensing methods.

The need for sustainable and efficient use of resources underscores the importance of sensing technologies that balance performance and cost-effectiveness. TiO_2 emerges as an exemplar in this regard, offering an auspicious path towards next-generation sensing platforms. Through rigorous scientific investigation and innovative

technological approaches, this research endeavor aims to unveil the latent sensing potential of TiO_2 for volatile liquid species, thereby catalyzing a transformative paradigm shift in the field of gas sensing.

Exploring the intricate world of TiO_2 -based gas sensing technology is not just about technological innovation – it's a profound quest to understand the fundamental mechanisms behind gas-solid interactions. By delving into the sensing behavior of TiO_2 towards volatile liquid analytes, this research endeavor aims to provide valuable insights into the molecular dynamics that govern these interactions. This knowledge can pave the way for the development of highly sensitive, selective, and reliable sensing platforms.

The investigation of TiO_2 -based gas sensors for volatile liquid detection converges the demands of scientific inquiry and societal needs. Through a judicious blend of scientific rigor, empirical investigation, and technological innovation, this research effort seeks to unravel the latent sensing potential of TiO_2 . The findings of this work have the potential to usher in a new era of gas sensing methodologies, with farreaching implications for environmental sustainability, industrial process optimization, and public health surveillance.

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CHAPTER 2 EXPERIMENTAL METHODS

EXPERIMENTAL METHODS

2.1 LabVIEW: LabVIEW is a user-friendly, graphical programming tool that allows even non-technical users to create algorithms and applications. Unlike traditional text-based coding, LabVIEW uses visual block connections to build programs, avoiding the risk of human errors in data collection and processing.

LabVIEW-based systems excel at taking measurements across multiple channels, as they can effectively interface with physical objects and systems. The software provides a wide range of virtual instruments (VIs) and functions for acquiring, analyzing, displaying, and storing data, as well as troubleshooting tools to help users debug their code.

LabVIEW is a powerful and integrated development environment suitable for various instrumental applications, from managing large, professional projects to integrating project management tools, graphical debugging tools, and standardized source code control.

LabVIEW is best-suited for developing code for automated test systems, systems used for validating designs and systems used to test products before they leave the factory. In Graphical programming, the syntax is knowledge but not required.

Advantages:

• User-friendly UI: It has a user-friendly drag and drop kind of interactive User Interface.

- Error Detection: Errors are indicated as we wire blocks in graphical programming while in Text-based programming, to check error, the program has to be compiled.
- Built-in Functions: It supports thousands of inbuilt functions that range from analysis and I/O etc. These belong to the function palette.
- Scalable: As LabVIEW has a modular design making it easy to scale and modular programs.
- Professional Development Tools: It has a plethora of tools that help integrate and debug large applications.
- Open environment: It has tools needed for many open environment developments.
- Object-oriented design: It supports object-oriented programming structures enabling encapsulation and inheritance to create modular and extensible code.
- Compiled language: Being a compiled language it is faster [1].

We have used LabVIEW to build a program to track and plot the characteristics of the thin film semiconductor sensor under free air and in the presence of different volatile organic vapors like Acetone and Ammonium Hydroxide. A laptop containing the software was connected the multimeter. A code was built to plot Current in Amperes against Time in seconds. The Visual Block connections or the code mentioned is given below.

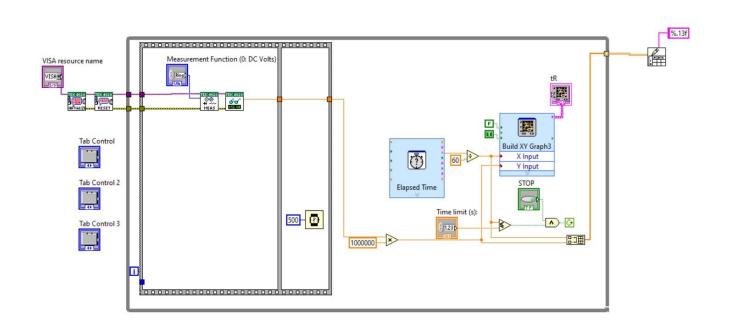


Figure 2.1: Graphical programming code

2.2 Spin coating: Spin-coating is an ideal method for applying a uniform thin coating onto a flat substrate. The substrate is secured onto a spinning stage, and then the coating material is applied to the centre of the substrate and allowed to spread evenly over the surface through centripetal acceleration forces.

- Speed and Time: The speed at which the material is spun and the duration of spinning time are crucial factors in controlling the thickness and consistency of the deposited film. Higher speeds typically result in thinner films, while longer spinning times can lead to more uniform coatings.
- Thickness Control: The thickness of the film can be adjusted by modifying factors such as the concentration of the solution, viscosity of the liquid, and speed of rotation. By optimizing these parameters, researchers can achieve precise control over film thickness.
- Uniform Coatings: Spin coating is known for its ability to produce highly uniform thin films with minimal defects or variations in thickness across the substrate surface.

- Wide Applications: Spin coating is widely used in various industries, including microelectronics, optics, and biotechnology, for applications such as photoresist deposition, dielectric layers, anti-reflective coatings, and organic electronics.
- Advantages: Some key advantages of spin coating include its simplicity, costeffectiveness, scalability for high volume production, and ability to coat a wide range of substrates with different shapes and sizes.
- Limitations: Despite its many benefits, spin coating does have some limitations. For instance, it may not be suitable for depositing thick films or materials with high viscosity due to challenges in achieving uniformity at greater thicknesses [2].

Overall, spin coating is a versatile and widely used technique for depositing thin films with excellent control over thickness and uniformity. Its simplicity and effectiveness make it an essential tool in many research and industrial applications.



Figure 2.2: Spektrospin- Spin coater

2.3 Sensor fabrication

Different methods used to spread the sample over the glass slide

Brush method:

Spreading samples evenly on microscope slides is crucial for effective analysis. The brush method is a technique that helps achieve this. It involves placing a small drop of the sample on the slide and using a fine-bristled brush to spread it evenly across the surface. The brush ensures the sample covers the entire slide without creating air bubbles or uneven patches. This method is particularly useful for viscous or sticky samples that are difficult to spread using other techniques. By providing precise control over the sample distribution, the brush method results in a more uniform and consistent slide preparation for microscopy analysis.

Drop and spread method:

In order to obtain a uniform spread of a sample of paste over a glass slide, a specific method can be employed. In the "drop and spread" technique, a small amount of the paste is placed onto the centre of the glass slide using a dropper or pipette. Once the paste is on the slide, a spreader or a glass rod can be used to evenly and gently spread the paste across the surface of the slide. This method ensures that the paste is distributed uniformly and covers the entire slide without any gaps or unevenness.

Spin coating method:

This paste can be uniformly applied to the slide using spin coating. Through both dynamic and static processes, we coat ZnO paste on to the glass plate. After a coat, the plate is dried in an oven at 150°C for 20 minutes. Several coating is done for desired

thickness. Later it is used for sensing.

2.4 Gas used for Gas sensing

Acetone vapor:

Acetone is a chemical that evaporates easily. It is used in many industrial processes and consumer products. Breathing in high levels of acetone vapors can be harmful to people's health. It can cause breathing problems, irritate the eyes and skin, and even affect the nervous system. Therefore, it is important to have a reliable system to detect and monitor acetone vapor levels in the environment.

Ammonium hydroxide:

Ammonium hydroxide, also known as ammonia water, is a compound that consists of ammonia dissolved in water. It is commonly used in various applications, including gas sensing. Ammonium hydroxide is a versatile compound that can be used to detect the presence and measure the concentration of various gases, such as LPG(liquefied petroleum gas) and ammonia vapours. This compound is often used in gas sensors due to its ability to react with certain gases and produce observable changes that can be detected. One example of the use of ammonium hydroxide in gas sensing is in the detection of LPG.

2.5 X-Ray diffraction

X-ray diffraction is a fundamental phenomenon arising from the interaction between X-ray radiation and the ordered atomic structure of crystalline materials. As X-rays encounter a crystal lattice, they undergo scattering due to the periodic arrangement of atoms within the crystal. These scattering results in the generation of an interference pattern, akin to the diffraction patterns observed when light interacts with a diffraction grating.

The Bragg law, a cornerstone principle in X-ray diffraction, elucidates the conditions under which constructive interference occurs between X-rays scattered from adjacent atomic planes within the crystal lattice. According to this law, constructive interference arises when the path length difference between two scattered X-rays is equal to an integer multiple of the X-ray wavelength. Mathematically expressed as $n\lambda=2d\sin(\theta)$, where n is an integer representing the order of diffraction, λ is the wavelength of the incident X-rays, d is the spacing between atomic planes, and θ is the angle of incidence, this relationship governs the formation of distinct diffraction peaks in X-ray diffraction patterns.

In the realm of materials science, X-ray diffraction (XRD) emerges as an indispensable analytical technique for elucidating various characteristics of crystalline materials. Through XRD analysis, researchers glean invaluable insights into the chemical composition, crystal structure, phase identification, and microstructural properties of diverse materials, ranging from bulk solids to nanomaterials. Moreover, XRD facilitates the characterization of crystallite size, lattice distortions, preferred orientation, and layer thickness, thereby enabling comprehensive assessment and understanding of material properties.

The historical lineage of X-ray diffraction traces back to the pioneering work of Wilhelm Röntgen, who serendipitously discovered X-rays in 1895. Röntgen's groundbreaking discovery laid the foundation for the subsequent development of X-ray technology and its multifaceted applications, including X-ray diffraction. By

harnessing X-rays with wavelengths commensurate to the interatomic spacings within crystalline materials, XRD enables the elucidation of intricate structural details and the discernment of crystalline phases in complex material systems.

In contemporary materials research, X-ray diffraction stands as a quintessential tool for unraveling the mysteries of crystalline matter, facilitating advancements across diverse scientific disciplines and industrial sectors. Through its non-destructive and precise interrogation of material structure and composition, XRD continues to catalyze innovation and drive progress in materials science and technology.

2.6 Materials And Apparatus Used

1. 5-1/2 digit multimeter:

The 5-1/2 digit multimeter is an advanced electronic device engineered for exceptional precision and accuracy in measuring a wide range of electricity quantities. The designation "5-1/2 digit" signifies the device's ability to display five whole digits along with an additional half-digit on its digital screen during measurements, providing unparalleled resolution and detail. The term "5 Digits" denotes the multimeter's capability to exhibit up to five characters on its display when conducting electrical measurements. Each digit is capable of representing numerals from 0 to 9 inclusively, allowing for a comprehensive range of readings spanning from 00000 to 99999. This expansive numerical display empowers users to obtain precise and detailed measurements across various electrical parameters with utmost clarity. Furthermore, the designation "1/2 Digit" introduces an additional level of precision beyond whole numbers. This half-digit typically appears in a position slightly beyond the fifth digit and is capable of representing values ranging from -1 to +1. While

subtle, this half-digit enhances the granularity of measurement results, enabling users to discern minute variations and fluctuations in electrical quantities with exceptional sensitivity and accuracy. In essence, the 5-1/2 digit multimeter stands as a pinnacle of measurement instrumentation, offering unparalleled precision, accuracy, and detail in the evaluation of electricity quantities [4].

Its advanced capabilities make it indispensable for a wide range of applications, from scientific research and engineering to industrial maintenance and troubleshooting, where precise measurement and analysis are paramount.

2. Dc regulated dual variable power supply:

In electronics laboratories and workshops, a DC regulated dual variable power supply is a critical tool as it avails for adjustable DC voltage outputs for supplying power and evaluating electronic circuits. For electronics enthusiasts and professionals, a dual variable DC power supply that is regulated offers multiple features that can cater for different testing and prototyping purposes. Having two channels of independent output enables individuals to power more than one circuit in an instance or even offer different voltages to separate components. When it comes to delicate electronic gadgets, stable voltage supply is important since it prevents alteration. For example, variable voltage controller comes in handy when there is need to regulate the power from this device in order to suit definite needs. By integrating current limiting functions, these prevent electrical overloads or shot circuits that would otherwise cause harm to their users during use or disrupt some equipment. Their hardware deploys models comprising of digital displays for real time monitoring purposes as well as control of output voltage going into it alongside current. Due to their small size coupled with reduced weights, it can be used in different locations ranging from research centres dealing with electronics as well as colleges among many other places where they exist. In general, this serves as compulsory property that must be owned by every person who deals with building up electric circuits as much it cannot operate without; this is because it is reliable durable, provides precise power in terms of measurement as well as flexible enough against all odds whenever power supply becomes not enough [5].

3. Connection clips with probe :

Probes with connection clips are important tools for tasks involving electronic testing and measuring. They are secure connections between measuring instruments like multimeters or oscilloscopes and test points Tests loops/or probes_ usually include a metal clip or clamp that is attached to a flexible wire lead which has a probe tip at the end. The metal clip or clamp is created to gracefully grip the test point hence maintaining a reliable connection during tests. It may come in different forms such as alligator clips, crocodile clips or spring-loaded clips for various test points as well as uses. These are usually coated/ insulated so that shorts do not occur accidentally while protecting fragile parts from being destroyed. The probe wire lead allows for flexibility of use so it may be placed precisely at the necessary points during readings. And to minimize interference between signals, reliable electric connection is guaranteed through contact between probes at test points, while ensuring minimum signal loss.

4. 500 ml beaker:

A focal element in a gas sensor unit set up is the 500 ml beaker which acts as a gas detection device sensor platform and a cause of containing volatile liquid used in detecting gases. Chosen for its compatibility with both sensing materials and the

targeted gas, the beaker shields sensitive compounds or coatings hence allowing them to be deposited and interacted with. The sensing material is placed inside the beaker by means of some twin or wires, which is placed exactly at the centre and a few centimetres above the base of the beaker resulting to increased detection sensitivity. Placing a volatile liquid onto the sensor materials was how the beaker was utilized to promote reaching the target gas molecules with an opportunity to interact effectively with sensing elements required for detection. To begin with, proper sealing has to be done in order to contain all of that volatile liquid inside its chamber thus eliminating any chances of pollution around us. Aluminium foil is used for sealing the chamber, which can be moved and replaced again when necessary.

5. Tarsons Accupippet t-10 (1-10 microliters):

The Tarson Accupippett T-10 is a precision micropipette meant for dispensing 1 to 10 microliters (μ l) volumes accurately. With its user-friendly design and intuitive controls, the Accupippett T-10 constitutes a dependable instrument in the lab for the precise oversight of liquids. Generally, people will find it easy to adjust volume using their thumb wheel or button that controls the volume adjusting mechanism. Good quality materials were used in making it; hence it has long life span because of its sturdiness. Moreover, the Accupippett T-10 model from Accumetric is sold in many variations that allow users to adjust height of the pop-out needles so as to conveniently purge them without filling up that whole part through which liquid or air can pass as well as choose different kinds of plastic cones for various samples. In addition, it features adjustable tip ejectors for convenient tip disposal and compatibility with disposable tips used for different sample types and volumes. It comes in different forms which come with various features like the ability to adjust the height of the popout needle so that it can be easily purged without filling all these areas where only

liquid or air may pass by selecting plastic cones made up of varying materials meant for different samples while others have been pre-filled with liquids typical for specific applications including one which contains antibiotics meant for plant tissue culture applications. Besides using the Tarson Accupippett T-10 in terms of analytical, clinical or research experiments purposes, one is likely to leverage it because of its precision [6].

6. Lab View software:

LabVIEW is a graphical programming environment created by National Instruments. It is useful across various fields like education, scientific research, and business operations. LabVIEW allows users to perform tasks such as data collection, instrument operation, analysis, and modeling. The program is based on a graphical programming technique that enables individuals to create applications by linking different functional blocks or nodes on a visual interface. These nodes represent various functions like user interface controls, data acquisition, and signal processing. By connecting these blocks in a flowchart-like manner, users can construct their own Virtual Instruments (VIs). LabVIEW's flexibility, scalability, and robustness make it a versatile tool used in fields like scientific research, engineering, education, and industrial automation. It enables users to quickly develop applications for a wide range of tasks, from simple data logging to complex control systems.

7. Glass plate:

To make sure that sensor performance is good, it is important that when you are using a glass plate to coat sensing material, this job has to be done using cautious steps to create a consistent and a binding film. In the beginning, the glass plate has to be cleaned thoroughly until there are no impurities which might affect the adhesion of the film. Composition of coating solution comes next; usually it has sensitive compounds or polymers which are termed as the sensing materials. Within this solution, there are many ways in which these solutions can be applied to the glass plate; such as spin and dip coating methods.

It is after this step that drying or curing process will be done for removing solvents as well as solidifying a coat on it. Here we are using a glass plate of length 3 cm and 1.5 cm of width. The glass plate of the desire dimensions can be cut using a diamond or glass cutter.

8. Other Accessories

- Brush: A 9mm width flat paint brush is used to coat the sensing materials to the glass plate. The smooth bristles help to give uniform and accurate strokes over the glass plate.
- •
- Aluminium foil: aluminium foil is used to cover the sensing chamber and the glass plate with the sensing material. The foil helps to prevent the escape of the volatile liquid for sensing. It also acts as a conductive layer around the glass plate for better conduction and a proper platform for clipping the connection clips with probe.
- Silver paste: Silver lines a drawn over the glass plate containing the sensing material. This helps in the conductivity and the process must be done with extreme care and accuracy. The silver powder can be made into a silver paste by using silver diluter.

• Ethanol: Ethanol is utilized as a diluting agent in the creation of the TiO_2 paste. Its high volatility allows it to vaporize easily, rendering it inert to the sensing material. Ethanol is considered an ideal agent for this process due to its properties.



Figure 2.3: 5-1/2 Digit Multimeter



Figure 2.4: Variable dual power supply

2.7 Sensor Samples

Sample 1: TiO₂

Titanium dioxide, also known as titanium (IV) oxide or titania, is the inorganic compound with the chemical formula TiO_2 .

Preparation:

To create a colloidal suspension for gas detection, grind approximately 0.5 grams of Nano crystalline titanium dioxide (TiO_2) in a mortar and pestle. Add a few drops of very dilute acetic acid, then alternate between grinding and adding more dilute acid until you obtain a smooth, paint-like consistency. Avoid making the mixture too thick, like toothpaste. Evenly apply this paste to a slide, and it can now be used for gas detection.



Figure 2.5: TiO2 sample

Sample 2: ZnO

Zinc oxide, also known Calamine or Zinc White. It is naturally found as a mineral zincite.

Preparation:

Grind about 0.5 grams of Nano crystalline titanium dioxide- (ZnO) using a mortar and pestle, adding a few drops of dilute- acetic acid as you go. Keep alte-rnating between grinding and adding the- acid until you get a smooth, paint-like colloidal suspension. Spre-ad this paste evenly ove-r a glass slide using a spin coating method, coating it through both dynamic and static processe-s. Dry the coated slide in an ove-n at 150°C for 20 minutes. Repeat the- coating process several time-s to achieve the de-sired thickness. The coate-d slide can then be use-d for sensing applications [3].



Figure 2.6: ZnO sample

CHAPTER 3 EXPERIMENTAL

EXPERIMENTAL METHODS

3.1 Fabrication Procedure for Metal Oxide Gas Sensors

Step 1: Preparation of Sensing Material

- Measure three spoons of titanium dioxide (TiO₂) powder and transfer it to a petri dish.
- Add three to four drops of ethanol incrementally to the TiO₂ powder while stirring until a paste-like consistency is achieved.
- Ensure thorough mixing to attain a homogeneous paste without clumps.
- The resulting TiO₂ paste serves as the sensing material for the gas sensor fabrication.

Step 2: Application of TiO₂ on Glass Plate

- Prepare a glass plate with dimensions of 3 cm length and 1.5 cm width.
- Clean the glass plate using a suitable cleaning agent followed by rinsing with ethanol and air drying.
- Apply the TiO₂ paste onto one side of the glass plate using a 9mm paintbrush, ensuring uniform distribution.
- Heat the glass plate in an oven to promote adhesion and consolidation of the TiO₂ paste.
- Apply a second layer of TiO₂ paste onto the treated surface of the glass plate and repeat the heating process.

Step 3: Annealing

- Subject the glass slide coated with TiO₂ to controlled heating in an oven at 473 Kelvin for 20 minutes.
- Allow the glass slide to cool gradually to ambient temperature over 10 minutes.
- Apply a second layer of TiO₂ onto the treated surface of the glass slide with precision and care.
- Ensure careful handling to prevent disruption or damage to the TiO₂ coating.

Step 4: Silver Electrode Fabrication

- Crush and refine silver powder to achieve a fine consistency.
- Dilute the finely powdered silver with a silver diluter solution to attain a uniform consistency.
- Use a 9mm paintbrush to apply two parallel silver conducting lines atop the TiO₂-coated glass substrate, maintaining a precise separation distance of 15mm.
- Encase each silver line individually with aluminum foil to optimize conductivity and mitigate interference.



Figure 3.1: Silver lined TiO₂ glass plate

3.2 Experimental Setup: Gas Sensing

In the experimental setup for metal oxide gas sensor testing, meticulous attention to detail is paramount to ensure the integrity and accuracy of the results obtained. The sensor, coated with TiO2 for gas sensing capabilities, is meticulously positioned within a pristine 500-milliliter beaker, meticulously cleaned to eliminate any potential contaminants that could compromise the experiment's outcomes. Positioned precisely at a predetermined height of 200 milliliters, the sensor's placement guarantees uniform exposure to the target environment, optimizing sensitivity and accuracy in detecting pertinent parameters. Fixed securely at the focal point of the beaker, the sensor's plain surface, adorned with the TiO2 coating, serves as the primary interface for interaction with the surrounding medium, facilitating precise measurement and analysis. Two connection clips affixed to the sensor terminals ensure a secure electrical interface for data acquisition and signal transmission, minimizing potential sources of error or variability. The experimental setup also involves meticulous circuit connections, with probes meticulously linked to essential instrumentation such as a DC power supply and a digital multimeter (DMM). Configured for precise voltage regulation and current measurement, the integrated setup, facilitated by a robust USB connection to a laptop computer, enables real-time data transmission and comprehensive analysis using advanced software tools like Lab VIEW

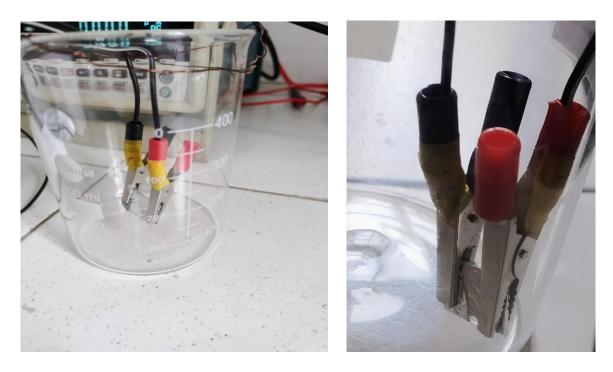


Figure 3.2: Gas sensing chamber

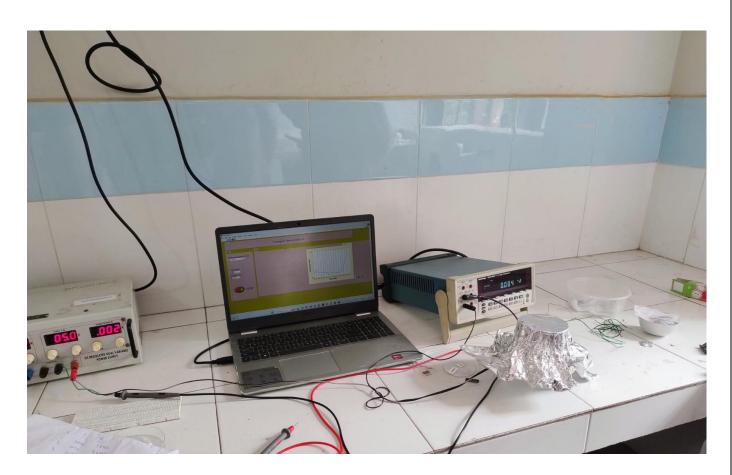


Figure 3.3: Sensing unit during the detection of ammonia

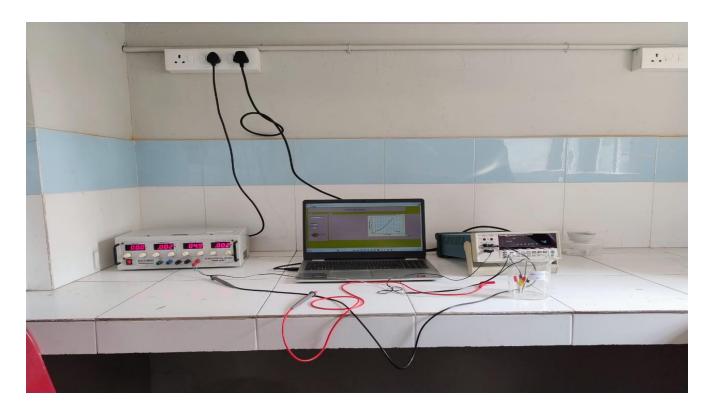


Figure 3.4: Sensing unit when exposed to air at room temperature

3.3 Experimental Protocol for TiO2-Based Sensor

In the experimental protocol focusing on TiO2-coated sensors, meticulous procedures are employed to investigate the sensor's response to volatile compounds, particularly targeting ammonia. The setup involves precise control of voltage parameters and systematic addition of volatile liquid samples to the sensing chamber, enabling comprehensive analysis of sensor response characteristics. Initially, the DC power supply is set to provide a standardized voltage of 5 volts, establishing a baseline for current measurements. The sensor, placed within the sensing chamber and exposed to ambient air, undergoes monitoring to ascertain the base current at room temperature over approximately 2 minutes. Subsequently, precise volumes of ammonia are incrementally introduced into the sealed sensing chamber using a T-10 micro pipette. The chamber is sealed with aluminum foil to control exposure, and fluctuations in

current are recorded over 8 minutes, with a discernible peak indicating heightened sensitivity to ammonia. After reaching a plateau, indicating saturation, the foil is removed, and a subsequent decline in current reflects dissipation of the ammonia vapor. Response and recovery times are calculated from the data, providing insights into sensor dynamics under varied experimental conditions. The protocol is iterated with adjustments in voltage parameters to discern their impact on sensor response and sensitivity to volatile compounds.

3.4 Aniline Method with Thermal Augmentation

In this iteration, thermal augmentation is introduced to expedite response dynamics. Initial response and recovery times are documented at ambient room temperature, serving as a baseline. The experimental paradigm evolves with controlled thermal stimuli, with the volatile liquid incrementally added accompanied by temperature elevation. A heater apparatus maintains the environment at approximately 313 Kelvin (K), while current variations are monitored to provide insights into sensor response under thermally enhanced conditions. The data offer a comprehensive depiction of temperature modulation's effects on sensor behavior and sensitivity, facilitating nuanced analysis and optimization of sensor technologies through thermal modulation.

3.5 Exploring CuO-Coated Sensors

A parallel experiment utilizing a spin-coated glass slide with copper oxide (CuO) is conducted, offering insights into alternative sensing materials' detection capabilities. Procedures mirror those of the TiO2-based experiment, with the CuO-coated sensor exposed to ammonia vapors to characterize response dynamics. Comparative analyses between materials elucidate the sensor's performance metrics and suitability for volatile liquid detection applications, contributing to sensor technology advancements.

3.6 Diverse Volatile Liquids: Acetone Detection

Expanding the experimental scope, the CuO-coated sensor's versatility is demonstrated by detecting acetone vapors. Employing the same setup and procedural protocols, the sensor exhibits heightened responsiveness to acetone, underscoring its suitability for multifaceted sensing applications. Comparative analyses between responses to acetone and ammonia further highlight the sensor's discriminatory capabilities and versatility, advancing sensor technology for diverse applications.

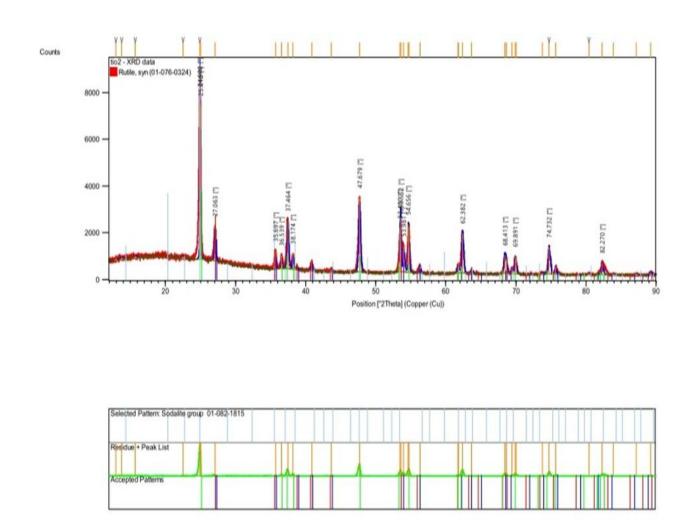
<u>References</u>

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CHAPTER 4 RESULS AND DISCUSSION



4.1 Structural Characterization of TiO₂ using XRD

Figure 4.1: XRD spectra of TiO₂ nano powders

Figure 4.1 shows the XRD spectrum of mixed-phase TiO_2 unveiled distinct peaks on the diffraction pattern, representing various crystallographic planes and suggesting the presence of multiple crystalline phases. Comparison of this experimental pattern with reference data from databases confirmed the coexistence of primary phases, notably anatase and rutile TiO_2 , alongside possible traces of other polymorphs. The observed peak positions and intensities closely matched the anticipated crystal structures of anatase and

rutile phases, thus affirming the reliability and precision of the XRD analysis [1].

Analysis of the XRD spectrum revealed distinct peaks at 2θ angles of approximately 25.3° and 47.4°, corresponding to the (101) and (200) crystallographic planes of anatase TiO₂, respectively [2]. Similarly, peaks at 2θ angles of around 27.5° and 36.1° were observed, corresponding to the (110) and (101) planes of rutile TiO₂, respectively [3]. The intensities of these peaks indicated a higher abundance of rutile TiO2 compared to anatase TiO₂ in the sample. Additionally, the sharpness of the peaks suggested a higher degree of crystallinity for rutile TiO₂ compared to anatase TiO₂, with narrower peaks indicative of larger crystallite sizes and higher structural order. These findings provide valuable insights into the phase composition and crystalline properties of the mixed-phase TiO₂ sample.

The quantification of anatase and rutile phases in the mixed-phase TiO_2 sample was determined through analysis of peak positions, intensities, and shapes in the XRD spectrum. The distinct peaks corresponding to different crystallographic planes were observed, indicating the presence of multiple crystalline phases within the sample. By using *Xpert High score* software Peak fitting and integration techniques were employed to quantify the relative abundance of each phase based on the intensity of their respective peaks. The quantification revealed that the sample predominantly consisted of rutile TiO₂, with a relative abundance of approximately 58.4%, while anatase TiO₂ constituted the remaining 41.6%. These findings were visually represented in a pie graph, illustrating the relative proportions of anatase and rutile phases in the sample.

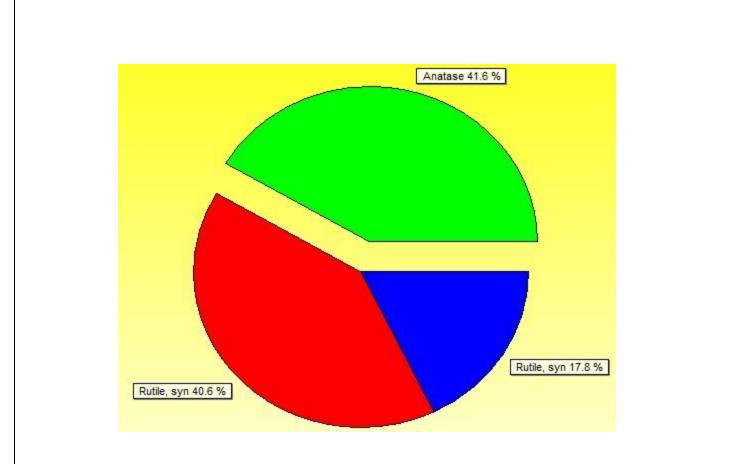
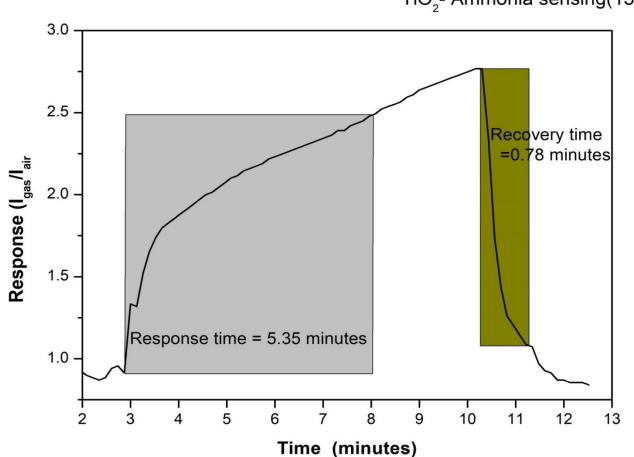


Figure 4.2: Relative abundance of rutile and anatase phases in the mixed-phase TiO2 sample, showing an approximately 58.4% predominance of rutile and 41.6% of anatase.

4.2 Gas sensing of TiO2 & ZnO



TiO₂- Ammonia sensing(15μL)

Figure 4.3: Response of TiO2 Gas Sensor to Ammonia Detection Over Time"

Based on the experimental data obtained during ammonia detection, the response and recovery times of the TiO_2 gas sensor were calculated. The response time refers to the duration taken by the sensor to detect and exhibit a change in its electrical conductivity upon exposure to ammonia vapor. From the experimental data, it was observed that the TiO_2 sensor demonstrated a response time of approximately 5.35 minutes, indicating the presence of ammonia. Conversely, the recovery time denotes the period required for the sensor to revert to its initial baseline conductivity levels after the removal of the ammonia vapor stimulus. The experimental results revealed a recovery

time of around 47 seconds for the TiO2 sensor, signifying its efficient restoration to baseline conditions once the ammonia vapor was eliminated. These response and recovery times highlight the dynamic and reversible nature of the TiO_2 gas sensor's interaction with ammonia vapor, showcasing its potential for real-time monitoring and detection applications in various industrial, environmental, and healthcare settings. It seems that the recovery time is much faster than the response time during the sensing of ammonia using TiO_2 .

4.3 Gas sensing of ZnO

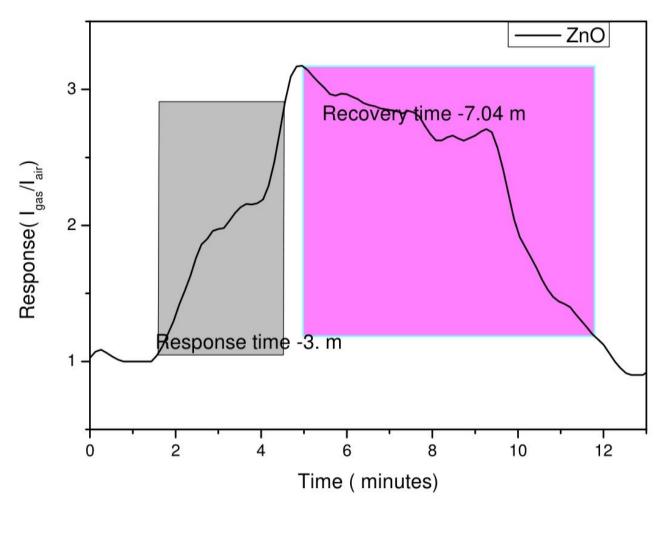


Figure 4.4: Response of ZnO Gas Sensor to Ammonia Detection Over Time"

When using ZnO as the sensing material, the presence of ammonia is detectable through temperature increases. However, experimental observations indicate that ammonia detection by ZnO is relatively weak. The response time for detection is approximately 3 minutes, while the recovery time to reach the initial current value at atmospheric air and room temperature is observed to be 7.04 minutes. Unlike TiO_2 sensing, ZnO exhibits a longer recovery time compared to its response time.

CHAPTER 6 CONCLUSION

This study comprehensively explored the construction and analysis of thin film metal oxide semiconductor (MOS) gas sensors, utilizing the capabilities of LAB View software for thorough data acquisition and analysis. Through a meticulously designed experimental framework, the researchers rigorously evaluated the performance characteristics of these sensors, including sensitivity, selectivity, response time and long-term stability.

The findings highlight the potential of thin film MOS gas sensors as reliable and versatile tools for gas detection and monitoring across various applications. By harnessing the unique properties of metal oxide semiconductors and leveraging LABView's robust platform for sensor control and data processing, this research has advanced our understanding of gas sensing technology and its practical implications.

A key strength of this research lies in its integration of LABView, which not only facilitates real-time monitoring and automation but also enables sophisticated data analysis and visualization. This holistic approach enhances the efficiency and accuracy of gas sensing systems and opens avenues for exploring complex gas interactions and phenomena.

The research on gas sensing technology has made significant advancements, but there are still challenges to address. Future work may focus on optimizing sensor design, exploring new materials, and improving selectivity towards specific gases.

Additionally, advancements in data processing techniques, combined with artificial intelligence and machine learning, offer exciting possibilities for enhancing the capabilities and functionalities of metal-oxide semiconductor gas sensors. By continuing to push the boundaries of innovation and collaboration, the potential of this technology can be realized in addressing critical societal and environmental issues.

In conclusion, the insights from this research highlight the transformative potential of thin-film metal-oxide semiconductor gas sensors in diverse applications, from environmental monitoring and industrial safety to healthcare. By integrating material science, electronics, and data analytics, we can develop safer, more sustainable and technologically advanced gas sensing solutions.

Future Scope

The integration of composite materials holds significant promise for enhancing gas sensor performance. By incorporating composite materials into thin-film metal oxide semiconductor (MOS) gas sensors, we can leverage the synergistic effects of multiple components to improve sensitivity, selectivity, and response time. The scope for composite materials encompasses a wide range of possibilities, including combining metal oxides with carbon-based materials, polymers, or nanoparticles. These composites can offer enhanced surface area, increased catalytic activity, and improved gas adsorption properties, leading to superior sensor performance. Furthermore, the tunability of composite materials allows for customization based on specific gas detection requirements, enabling the development of sensors tailored to different applications and environments. Through further research and development in composite materials, we can unlock new opportunities for advancing gas sensing technology and addressing complex challenges in environmental monitoring, industrial safety, and healthcare.

<u>Reference</u>

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APPENDIX A

Name and formula

Reference code:	01-076-0324
Mineral name:	Rutile, syn
ICSD name:	Titanium Oxide
Empirical formula:	O ₂ Ti
Chemical formula:	TiO ₂

Crystallographic parameters

Crystal system: Space group:	Tetragonal P42/mnm
Space group number:	136
a (Å):	4.6452
b (Å):	4.6452
c (Å):	3.0017
Alpha ("):	90.0000
Beta ("):	90.0000
Gamma ("):	90.0000
Calculated density (g/cm^3):	4.10
Volume of cell (10 ⁶ pm ³):	64.77
Z:	2.00
RIR:	3.20

Status, subfiles and quality

Status:	Diffraction data collected at non ambient temperature
Subfiles:	Inorganic
	Mineral
	Alloy, metal or intermetalic
	Corrosion
	Modelled additional pattern
Quality:	Calculated (C)

Comments

ICSD collection code:	033844
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References

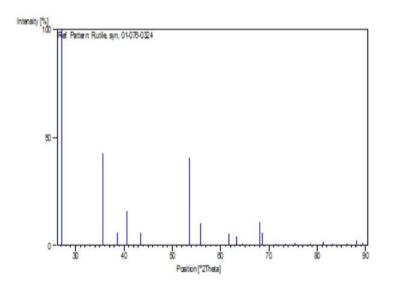
Primary reference:	Calculated from ICSD using POWD-12++, (1997)
Structure:	Sugiyama, K., Takeuchi, Y., Z. Kristallogr, 194 , 305, (1991)

Peak list

h	k	1	d [A]	2Theta[deg]	I [%]
1	1	0	3.28465	27.126	100.0
1	0	1	2.52113	35.581	42.9
2	0	0	2.32260	38.738	6.0
1	1	1	2.21582	40.686	16.0
	h 1 2 1	h k 1 1 1 0 2 0 1 1	h k 1 1 1 0 1 0 1 2 0 0 1 1 1	1 1 0 3.28465 1 0 1 2.52113 2 0 0 2.32260	1 1 0 3.28465 27.126 1 0 1 2.52113 35.581 2 0 0 2.32260 38.738

5	2	1	0	2.07740	43.530	5.8
6	2	1	1	1.70821	53.608	40.6
7	2	2	0	1.64233	55.943	10.2
8	0	0	2	1.50085	61.760	5.5
9	3	1	0	1.46894	63.255	4.5
10	2	2	1	1.44077	64.640	0.3
11	3	0	1	1.37610	68.080	10.6
12	1	1	2	1.36510	68.705	5.8
13	3	1	1	1.31942	71.439	0.5
14	3	2	0	1.28835	73.439	0.2
15	2	0	2	1.26057	75.334	1.1
16	2	1	2	1.21657	78.569	0.6
17	3	2	1	1.18391	81.180	1.5
18	4	0	0	1.16130	83.105	1.0
19	4	1	0	1.12663	86.270	0.4
20	2	2	2	1.10791	88.098	2.6
21	3	3	0	1.09488	89.425	1.2

Stick Pattern



APPENDIX B

Name and formula

Reference code:	01-071-1169
Mineral name:	Anatase
ICSD name:	Titanium Oxide
Empirical formula:	O ₂ Ti
Chemical formula:	TiO ₂

Crystallographic parameters

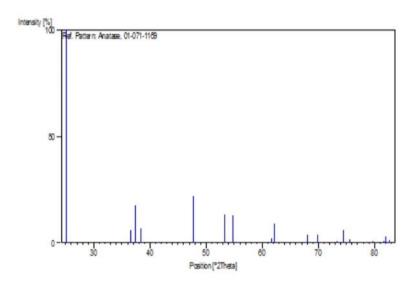
Crystal system: Space group: Space group number:	Tetragonal I41/amd 141
a (Å):	3.8040
b (Å): c (Å):	3.8040
	9.6140
Alpha ("):	90.0000
Beta ("):	90.0000
Gamma ("):	90.0000
Calculated density (g/cm^3):	3.81
Measured density (g/cm^3):	3.87
Volume of cell (10 ⁶ pm ³):	139.12
Z:	4.00
RIR:	4.80

Status, subfiles and quality

Status: Subfiles: Quality:	Diffraction data collected at non ambient temperature Inorganic Mineral Alloy, metal or intermetalic Corrosion Modelled additional pattern Calculated (C)
<u>Comments</u>	
Sample source: ICSD collection code: Test from ICSD:	Specimen from Binntal, Wallis, Switzerland. 009855 Calc. density unusual but tolerable.
References	
Primary reference: Structure:	Calculated from ICSD using POWD-12++ (1997) Horn, M., Schwerdtfeger, C.F., Meagher, E.P., Z. Kristallogr., Kristallgeom., Kristallphys., Kristallchem. 136 , 273, (1972)
<u>Peak list</u>	
No. h k l	d [A] 2Theta[deg] I [%]

1	1	0	1	3.53718	25.156	100.0
2	1	0	3	2.45087	36.637	5.9
3	0	0	4	2.40350	37.385	17.6
4	1	1	2	2.34733	38.314	6.9
5	2	0	0	1.90200	47.782	21.8
6	1	0	5	1.71604	53.344	13.3
7	2	1	1	1.67518	54.752	13.0
8	2	1	3	1.50261	61.680	2.3
9	2	0	4	1.49149	62.191	9.2
10	1	1	6	1.37659	68.052	4.0
11	2	2	0	1.34492	69.884	4.0
12	1	0	7	1.29181	73.210	0.4
13	2	1	5	1.27411	74.397	5.9
14	3	0	1	1.25711	75.578	1.6
15	0	0	8	1.20175	79.730	0.3
16	3	0	3	1.17906	81.584	0.4
17	2	2	4	1.17367	82.039	2.8
18	3	1	2	1.16695	82.615	1.3

Stick Pattern



No.	Visible	Ref. Code	Compound Na	Chemical Formula	Score	Scale F	SemiQu
	~	01-076-0324	Rutile, syn	Ti O2	35	0.206	41
	~	01-077-0444	Rutile, syn	Ti O2	26	0.096	18
	3 🔽	01-071-1169	Anatase	Ti O2	28	0.316	42

