SYNTHESIS AND CHARACTERIZATION OF CuO THINFILM

A PROJECT REPORT

Submitted by

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To

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In partial fulfilment of the requirements for the award of the Degree Of Bachelor of Science In

Physics

Department of Physics

Bharata Mata College, Thrikkakara

DECLARATION

I undersigned hereby declare that the project report"SYNTHESIS AND

CHARACTERISATION OF CuO THIN FILM", submitted for partial fulfilment of the requirements for the award of degree of Physics of the Mahatma Gandhi University, Kerala is a bonafide work done by me under supervision of Anu Philip , Asst. Professor, Dept. Of Physics. This submission represents my ideas in my own words and where ideas or words of others have been included; I have adequately and accurately cited and referenced the original sources. I also declare that I have adhered to ethics of academic honesty and integrity and have not misrepresented or fabricated any data nor idea or fact or source in my submission. I understand that any violation of the above will be a cause for disciplinary action by the institute and/or the University and can also evoke penal action from the sources which have thus not been properly cited or from whom proper permission has not been obtained. This report has not been previously formed the basis for the award of any degree, diploma or similar title of any other University.

Place: Kochi

CERTIFICATE

This is to certify that the report entitled SYNTHESIS AND CHARACTERISATION OF cuo THIN FILM submitted by SREELAKSHMI JITHESH AND VYSHNAVI MB to the Mahatma Gandhi University in partial fulfilment of the requirements for the award of the Degree of Bachelor of Science in Physics is a bonafide record of the project work carried out by him/her under my/our guidance and supervision. This report in any form has not been submitted to any other University or Institute for any purpose.

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ABSTRACT

This project focuses on the synthesis and characterization of copper oxide (CuO) thin films via the spin coating method for gas sensing applications. The thin films were deposited on a suitable substrate and characterized using various techniques including X-ray diffraction (XRD). The XRD analysis revealed the crystalline structure of the CuO thin films. Gas sensing properties of the thin films were investigated, demonstrating their potential for detecting specific gases. This study provides valuable insights into the fabrication and application of CuO thin films for gas sensing devices, contributing to advancements in sensing technology.

CONTENT

CHAPTER 1

INTRODUCTION

1.1 NANOPARTICLE COATING

Nanoparticle coating, a versatile technique, entails depositing nanoparticles onto a solid surface in multiple layers using various methods like the Langmuir-Blodgett method, spin coating, dip coating, and others. These methods offer precise control over nanoparticle arrangement and thickness, crucial for tailoring functionalities in diverse applications. In industries ranging from electronics to biomedicine, nanoparticle coatings play a pivotal role. For instance, in electronics, they can enhance the durability of surfaces and impart specific properties like conductivity, crucial for the performance of electronic devices. In biomedicine, nanoparticle coatings can be utilized for drug delivery systems, enhancing the efficacy and specificity of medication delivery to target tissues or cells. Moreover, nanoparticle coatings contribute to the development of advanced materials and devices with tailored performance characteristics. Whether it's creating super hydrophobic surfaces for self-cleaning applications or engineering biocompatible coatings for medical implants, the controllability and versatility of nanoparticle coatings drive innovation across industries.

1.2 SPIN COATING

The process of spin coating is used to apply homogeneous thin coatings to flat substrates. Typically, a little amount of liquid coating material is applied to the center of the substrate, which is either not spinning at all or spinning at a very slow pace. Centrifugal force is then used to spread the coating material throughout the substrate by rotating it. A spin coater, or just spinner, is a device used for spin coating. Until the required film thickness is reached, the fluid is rotated while spinning off the substrate's edges. Usually volatile, the applied solvent evaporatively disappears at the same time. The layer gets thinner as the angular speed of rotation increases. The solvent, as well as the solution's concentration and viscosity, affect the film's Spin coating is a commonly employed technique in the microfabrication of functional oxide layers on glass or single crystal substrates by the use of sol-gel precursors. This

process yields consistent thin films with nanoscale thicknesses. Advantage of spin coating: When creating CuO thin films, the spin coating technique offers a number of benefits. It enables the rapid and simple deposition of homogeneous, uniform films with excellent homogeneity and repeatable thickness. Its versatility lies in its ability to cover inorganic, organic, and inorganic/ organic solution combinations.
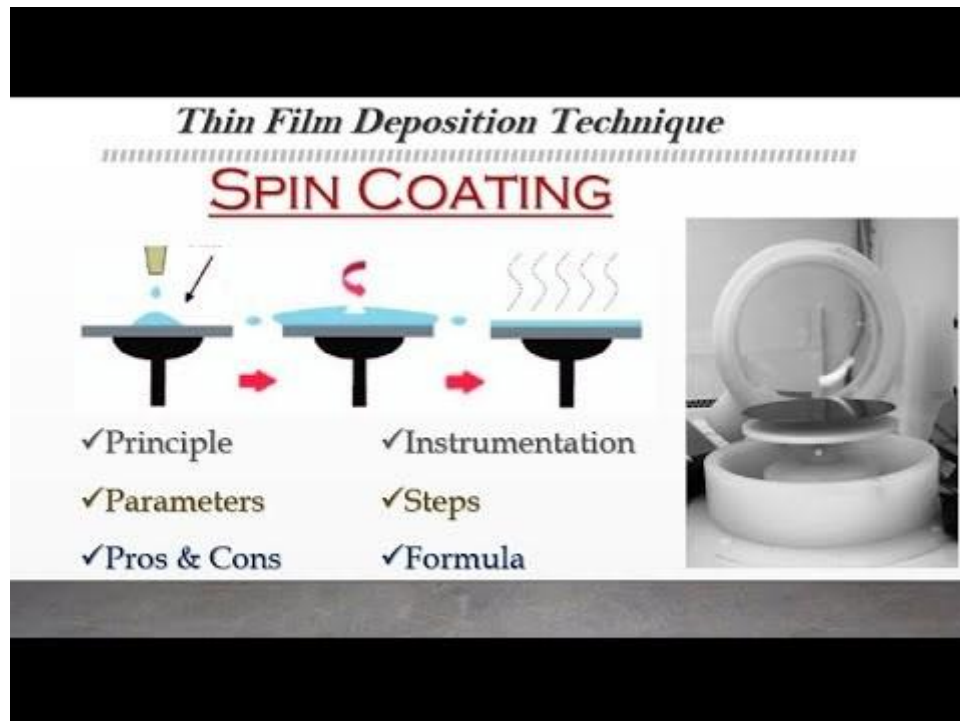

FIG 1.1

1.3 THIN FILM

A thin film is a layer of material with a thickness that can range from several micrometers to fractions of a nanometer. Various deposition techniques are employed by numerous industries to produce thin films. Many materials, particularly optical elements, have their surfaces covered with thin films to prevent wear, scratches, fingerprints, and even corrosion. Understanding the thickness, composition, stress state, shape, adhesion, degree of crystallinity, and any other relevant physical properties is necessary for a thorough characterisation of thin film It is noteworthy that although a plethora of characterisation tools are available for bulk materials, their applicability to the study of films is not universal because of the restricted sample volume that is accessible. Better methods for examining elements such as film composition are necessary since ceramic coatings are frequently made of three or more elements, and even small changes in stoichiometry can significantly affect the final product's qualities. For instance, by adjusting lattice characteristics

and transition temperatures, the deposition procedures used to create films can have a significant impact on the final properties. Therefore, it is not always possible to determine the composition of solid solutions using conventional methods (such as Vegard's law). Many technological advances in fields including magnetic recording media, electrical semiconductor devices, integrated passive devices, LEDs, and optical coatings have been made possible by developments in thin film deposition.
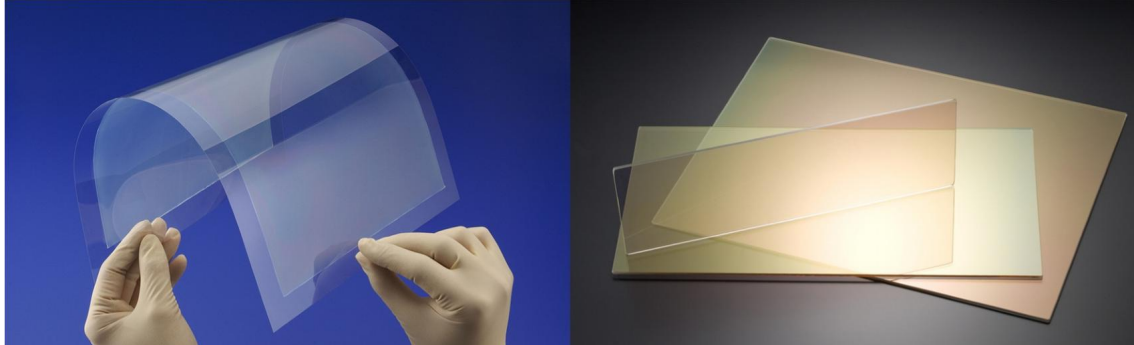
Fig 1.2

fig 1.3

1.4 THIN FILM GAS SENSORS

Thin film gas sensors are devices that detect the presence of specific gases in the environment through changes in electrical conductivity or other properties of thin films deposited on a substrate. These sensors are used in various applications, including environmental monitoring, industrial safety, and medical diagnostics. They offer advantages such as high sensitivity, low power consumption, and fast response times. Common materials used in thin film gas sensors include metal oxides like tin oxide and titanium dioxide, as well as polymers and carbonbased materials.
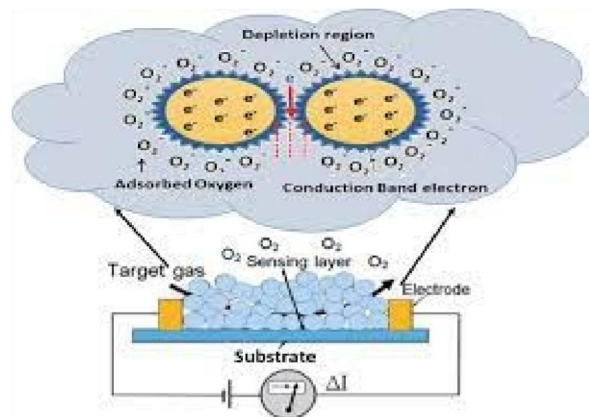
Fig 1.4

1.5 OBJECTIVE

project aims to create a room-temperature ammonia gas sensor using CuO films spin-coated onto glass substrates. This sensor seeks to accurately detect low concentrations of ammonia gas in ambient conditions, vital for environmental monitoring, industrial safety, and indoor air quality control. By leveraging CuO as the sensing material and the spin-coating technique, you're enhancing sensitivity, response time, and stability. The focus on cost-effectiveness and efficiency underscores practical implementation. This endeavor not only advances gas sensing technologies but also offers potential solutions for real-world challenges, fostering safer environments and improved air quality across various sectors.

CHAPTER 2

MATERIAL AND METHODS

2.1. COPPER OXIDE (CuO)

Another name for copper oxide (CuO) is cupric oxide. Another stable copper oxide that goes by the name cuprous oxide is Cu2O. Cuo is a low-cost, naturally abundant, and essentially non-toxic P-type semiconductor material. The majority of visible light may be absorbed by CuO film, which has band gap values between 1.2 and 2.1 eV and good photovoltaic performance. Consequently, it can be applied to thin-film solar cells as the absorber layer. The powder form of CuO is black to brown.79.545 g/mol is its molar mass. CuO is insoluble in water and has a low toxicity. Powdered Cuo With lattice constants of a = 4.6837, b = 3.4226, c = 5.1288, α = 90°, β = 99.54°, and γ = 90°, the crystal structure of CuO is monoclinic. Cuo structure CuO's potential for application in solar cell technology has recently attracted a lot of attention in thin-film technology. Several methods of deposition, such as filtration, thermal oxidation, and thermal evaporation.
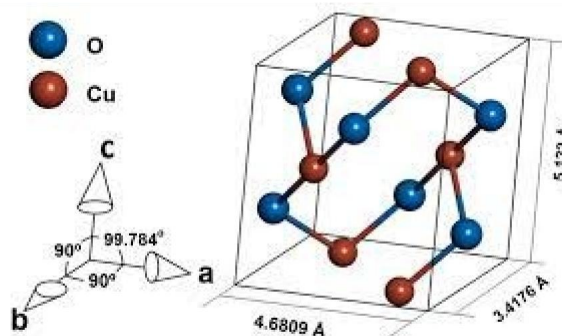
| Fig 2.1 | fig 2.2 |

2.2. GAS SENSING MECHANISM.

improved operational efficiencies.Copper oxide (CuO) is a material that senses gases at room temperature by changing how well it conducts electricity when they're around. It's really good at noticing ammonia gas. Scientists are trying different ways to make sensors with CuO so they can find gases better without needing high heat.Spin coating is a versatile and cost-effective method for depositing thin CuO films onto surfaces. It offers precise control over thickness, uniformity, and structure, ideal for

making high-performance gas sensors. Researchers have successfully used this technique to create sensitive and selective sensors for detecting ammonia at Advancements in room-temperature gas sensors are pivotal as they offer a significant leap forward in efficiency and applicability across diverse industries. Traditional gas sensors often necessitate high operating temperatures for precise detection, which can be both energyintensive and logistically challenging. By contrast, the development of sensors capable of accurately detecting gases like ammonia at ambient temperatures represents a breakthrough. Such sensors not only enhance safety protocols but also mitigate energy consumption and reduce associated maintenance costs. Furthermore, their deployment enables real-time monitoring in environments where conventional high-temperature sensors may be impractical or cost-prohibitive, thereby opening doors to novel applications and room temperature, promising advancements in gas detection technology. Now a days thin film gas sensors are used which is basically a class of chemiresistive sensor. A chemiresistive sensor is the one which changes its electrical resistance when there is a change in the nearby chemical environment. Chemiresistive sensors are class of chemical sensor that rely on the direct chemical interaction between analyte and the sensing material. This chemical interaction between the analyte and the sensing material can be by hydrogen bonding, covalent bonding or molecular recognition. There are several different materials which shows chemiresistive properties: Semiconducting Metal oxides, conductive polymers, and nanomaterials like graphene, carbon nanotubes, some nanoparticles. Transition metal dichalcogenides (TMD's).[1]

Chemiresistor Gas Sensor

- Chemiresistor gas sensors detect gas molecules by measuring changes in electrical resistance
- These sensors, often employing metal oxides like tin dioxide or zinc oxide, react to specific gases, altering their resistance
- Performance factors include the sensing material, its morphology, operating temperature, and environmental conditions
- Despite advancements from nanotechnology and materials science, challenges like selectivity in the presence of interfering gases remain
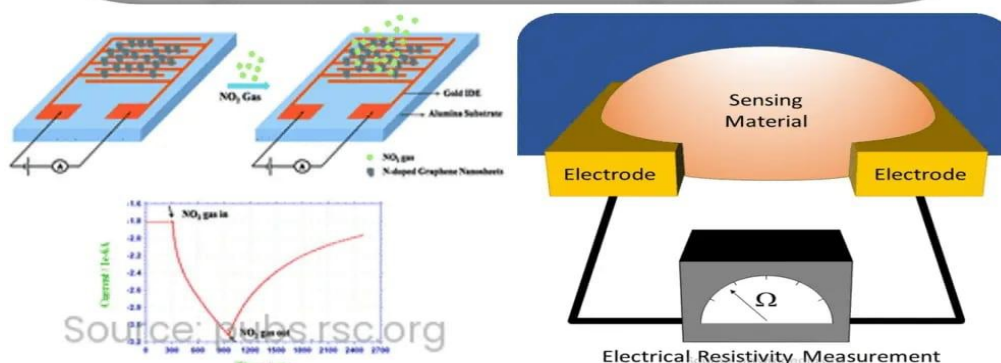- Future progress is expected as the technology and materials evolve

fig 2.3

In the case of gas sensing, CuO thin film-based sensors are particularly alluring due to their chemical stability, largewater contact angle (>90○), high sensitivity, faster response time, lower operating temperature, as well as proven ability to detect diverse gases such as NO2, H2, CO, and CO2.[2]

2.3. XRD TECHNIQUE

A very flexible method, X-ray diffraction (XRD) yields chemical data for phase analysis as well as elemental analysis. XRD is very helpful for texture analysis and stress measurements in addition to chemical characterisation. XRD analysis requires crystalline samples, although it can also determine a polymer's degree of crystallinity. Although XRD has historically been used to analyse bulk samples, new optical techniques have made it possible to use XRD for thin film examination as well. The method makes advantage of Bragg's law of diffraction.   The XRD technique is mainly used for phase identification of corrosion products in the oil and gas industry. For example, XRD effectively differentiates between iron oxides (FeO, Fe2O3, and Fe3O4) and oxyhydroxides (α-FeOOH, β-FeOOH, γ-FeOOH). Since the XRD technique identified phases based on their crystal structures, the technique cannot separate Fe2O3 and Fe3O4 due to the fact that both these phases have a similar crystal structure and very close lattice parameter. The author has shown the

application of Fourier transform infrared spectrophotometry (FTIR) in successful identification of all iron oxides (including Fe2O3 and Fe3O4) and oxyhydroxides. Calcite and other Ca containing phase formation on ferrous alloys used in oil exploration projects can be identified using XRD. In addition, XRD can be used in crystalline phase analysis of concrete and related materials. Typical XRD pattern of iron oxide is shown in figure The peaks in the XRD spectrum belong to Fe3O4 phase for different crystallographic planes identified as (hkl) with their corresponding dspacing (d) values in Å.[3]

CHAPTER 3

EXPERIMENT

3.1. SUBSTRATE PREPARATION

The process for cleaning glass plates in preparation for thin film creation involves several meticulous steps to ensure the surface is free from debris and contaminants. Initially, standard soda lime glass microscope slides are employed as the substrate. The cleaning procedure begins with the use of water and detergent to remove any visible impurities and debris from the surface of the glass plates.

Following the initial washing step, the glass plates undergo ultrasonic cleaning to further dislodge any stubborn particles and ensure thorough cleanliness. This involves placing the glass plates in a beaker of hot water within an ultrasonic cleaner and allowing it to run for a specified duration, typically 180 seconds. The ultrasonic vibrations help to agitate the cleaning solution, enhancing its ability to penetrate and clean even tiny crevices on the glass surface.

After ultrasonic cleaning, the glass plates are wiped with acetone, ethanol, and methanol to eliminate any remaining residues and ensure a pristine surface. These solvents are effective in removing organic contaminants and drying quickly without leaving behind any residue. Subsequently, the cleaned glass plates are returned to distilled water and subjected to another round of ultrasonic cleaning to ensure complete removal of any remaining contaminants or solvent residues. To further eliminate any moisture content, the glass plates are heated using a magnetic stirrer. This step helps to evaporate any residual water present on the surface of the glass, leaving behind a dry and pristine substrate ready for the deposition of thin films. Overall, this comprehensive cleaning process ensures that the glass plates are free from impurities and contaminants, providing an ideal surface for the deposition of high-quality thin films.

3.2. PRECURSOR PREPARATION

The procedure outlines a meticulous process for creating a solution with copper acetate monohydrate[Cu(CH3COO)2H2O] as the primary component. The method involves a series of sequential steps, each contributing to the formation of the final product. The following substances were used: ethylene glycol [C6H6O2], isopropanol [C3H7OH], and diethanolamine (DEA) [C4H11NO2],as the coating materials, solvent, and stabilizer. Initially, 9 ml of isopropanol[C3H7OH] and 0.5 ml of diethanolamine (DEA) [C4H11NO2]were employed to dissolve 699g of copper acetate powder, yielding a solution with a concentration of 0.35M. This step ensures the complete dissolution of the copper acetate, crucial for uniformity in the final solution. Subsequently, 0.5 ml of ethylene glycol [C6H6O2] was introduced into the precursor solution, presumably to serve as a coating material or additive, enhancing the properties of the solution.Following the addition of ethylene glycol, the mixture underwent agitation using a magnetic stirrer at 500 rpm for 120 minutes at room temperature. This step is essential for achieving homogeneity within the solution, facilitating the interaction between its components and promoting the formation of the desired product. The agitation process ensures thorough mixing and distribution of the substances, contributing to the consistency and quality of the final solution.The resultant solution exhibited a distinct dark blue tone, indicating the successful formation of the desired compound without any particle suspension. This characteristic suggests that the solution achieved a high level of uniformity and stability, meeting the desired specifications. Finally, the solution underwent filtration through a Polytetrafluoroethylene (PTFE) filter with a pore size of 0.45 µm. Filtration serves to remove any remaining impurities or particulate matter, ensuring the purity and clarity of the final product. In summary, the outlined procedure demonstrates a systematic approach to producing a solution with copper acetate monohydrate, incorporating precise measurements, controlled conditions, and thorough processing steps to achieve a high-quality end product.

fig 3.1

3.3. SPIN COATING OPTIMIZATION

A spin coater is used for the spin coating method. The solution is suspended on the glass surface due to the centrifugal force. The thickness of the film is depends upon

speed of spin coater, surface tension of the glass and viscosity of the solution.

Fig 2.2

3.3.1. PROGRAMME SETTING IN A SPIN COATER.

There are 4 segments to the spin coating process, and we have control over the spin coater's speed and duration for each part. DLL is deceleration, which aids in slowing down the spin coater, and ACC is acceleration, which this option helps to accelerate.

In the first segment, it is accelerating at 510 speed, and it will continue for up to 15 seconds.

fig 2.3

fig 2.4

fig 2.5

Following the initial segment, the system seamlessly transitions into the second segment, where it accelerates to a speed of 2500 for a duration of 45 seconds. This acceleration phase is integral to the overall operation, optimizing performance and efficiency for the subsequent stages of the process.

Fig 2.6

fig 2.7

fig 2.8

It will enter the third phase after the second, which will cause a 30-second deceleration.

Fig 2.9

fig 2.10

After the deceleration phase, the system advances to segment 4, marking the program's conclusion. This segment likely involves finalizing tasks for program completion.

Fig 2.11

3.3.2. THIN FILM FABRICATION

There are 2 types of spin coating static and dynamic spin coating. A small amount of solution of the coating material is dropped onto the center of the substrate. Then, the substrate is spun at high speed for a certain time (a few seconds) to spread the coating material uniformly on the substrates. This process is known as static spin coating[4]. In dynamic spin coating, a small amount of substrate is dropped onto the center of the surface when it is rotating. Once the program is configured, insert the glass plate into the spin coater's center and attach the spin coater to the vacuum pump. The spin coating was completed after the solution was poured into the glass. Five drops of the solution were added to the glass every fifteen seconds.The layer is heated to 200°C once it has formed. The glass was placed in a furnace and heated to 600°C once the same four layers had formed.

Fig 2.12

3.4. DEVELOPMENT OF GAS SENSORS

In the gas detection process, the volatile liquid ammonium gas is a key component, chosen for its ability to efficiently detect gases. This process typically occurs at room temperature, ensuring practicality and ease of operation. To create the gas sensor, conductive silver lines are meticulously drawn on the ends of a glass plate, forming the necessary circuitry for detection. The application of silver paste, derived from dissolving silver in a suitable diluter, facilitates the creation of these lines, ensuring conductivity and reliability. To enhance the sensor's performance, aluminum foil covers both ends of the glass plate, providing protection and stability to the sensor setup. The probe is configured with its positive end linked to a dual power supply and its negative end connected to a multimeter. This setup enables the measurement of current flow through the sensor. Maintaining a constant voltage from the dual power supply ensures consistency and accuracy in the measurement process. Once the sample under investigation comes into contact with the ammonium gas, the sensor's response is recorded, typically in the form of a plotted graph. This graph provides valuable insights into the presence and concentration of gases in the sample, serving as a critical tool in various industries such as environmental monitoring, industrial safety, and chemical analysis.

CHAPTER 4

RESULT AND DISCUSSION

4.1.STRUCTURAL CHARACTERIZATION OF CUO USING XRD

Measure the diffraction angles (2θ) and intensities of X-rays diffracted by a material using an X-ray diffractometer. Determine which peaks in the XRD pattern represent the planes of the crystal lattice. For each peak, find the interplanar spacing (d) using Bragg's equation (nλ = 2d sin θ), where μ is the diffraction angle, n is the order of the diffraction peak, and λ is the wavelength of the X-rays (typically known). Plot sin^2(θ) against intensities. Fwhm is calculated from the graph. The grain and lattice parameters are calculated from the given data.

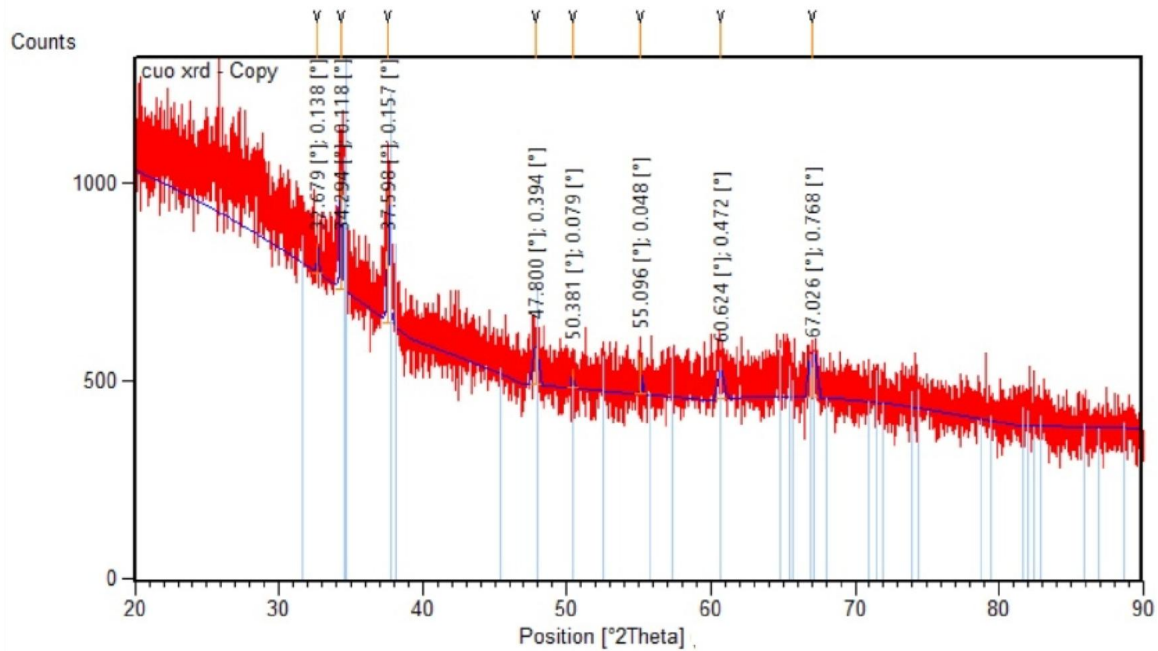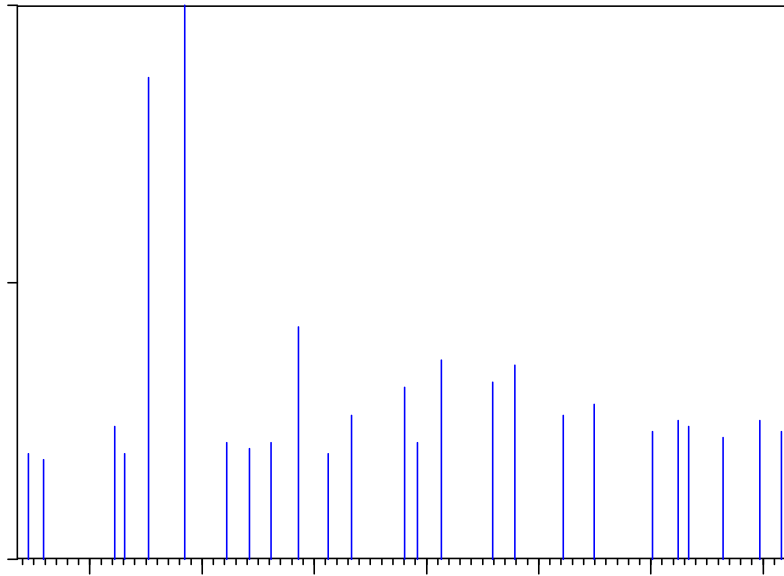fig 4.1

fig 4.2

A graph is plotted using the data that was obtained (Fig. 4.1). fwhm is then acquired. The Xray diffraction (XRD) pattern depicted in Figure 1 offers insights into the crystalline structure of the spin coated CuO thinfilms. XRD is a pivotal technique utilized for elucidating the atomic arrangement within crystalline materials by analysing the diffraction pattern of X-rays interacting with the lattice structure of the sample.

In the XRD pattern, the peaks signify the intensity of X-ray diffraction at specific angles (2θ), denoting the presence of crystalline planes within the CuO thinfilms. These peaks are indexed based on their positions and intensities, furnishing details about the crystallographic orientation and phase of the material.

The prominent diffraction peaks appearing around 35° and 38.6° correspond to reflections from the (-111) and (111) crystallographic planes of monoclinic phase CuO, respectively. These peak positions align with established values for CuO as documented in the "JCPDS" (Joint Committee on Powder Diffraction Standards) database, validating the monoclinic crystal structure of the synthesized CuO nanoparticles.

Significantly, the absence of X-ray diffraction peaks associated with other phases underscores the purity of the CuO films affirming its monoclinic phase composition.

Moreover, lower intensity peaks observed at various angles (e.g., 32.5°, 48.5°, 58.16°, etc.) correspond to reflections from diverse crystallographic planes, such as (100), (-202), (-202), (020), (202), (-113), (-311), (113), and (311). These peaks offer supplementary insights into the crystal lattice orientation and symmetry of the CuO films

No.	Pos. [°2Th.]	d-spacing [Å]	FWHM [°2Th.]
1	32.6791	2.74034	0.1378
2	34.2944	2.61487	0.1181
3	37.5976	2.39239	0.1574
4	47.7998	1.90289	0.3936
5	50.3809	1.81129	0.0787
6	55.0960	1.66554	0.0480
7	60.6237	1.52750	0.4723
8	67.0263	1.39515	0.7680

Table 4.1

The crystal grain size can be quantitatively calculated by Scherrer equation according to the diffraction peak broadening in the XRD curves. Actually, the results calculated by the Scherrer equation are the thickness that perpendicular to the crystal planes. [5]

B(2θ) $= K\lambda/$**L·cosθ**

using this equation grain is calculated for each value of 2 theta which is tabulated in the table 4.2 the average grain is

| No.	Pos. [°2Th.]	d-spacing [Å]	FWHM [°2Th.]	grain
1	32.6791	2.74034	0.1378	62.77

2	34.2944	2.61487	0.1181	73.56
3	37.5976	2.39239	0.1574	55.71
4	47.7998	1.90289	0.3936	23.07
7	60.6237	1.52750	0.4723	20.36
8	67.0263	1.39515	0.7680	12.96

Table 4.2

4.2. GAS SENSING PROPERTIES OF CuO

When using gas detection at room temperature, ammonium gas is utilized. The graph is created. The ratio of the current in the air to the current in the gas yields the response time. Response time is the amount of time that passes after gas is purged into the gas chamber before the sensor resistance reaches a specific percentage of its equilibrium value. Recovery time is the amount of time it takes a sensor to reach its baseline value following the measured variable's step removal.
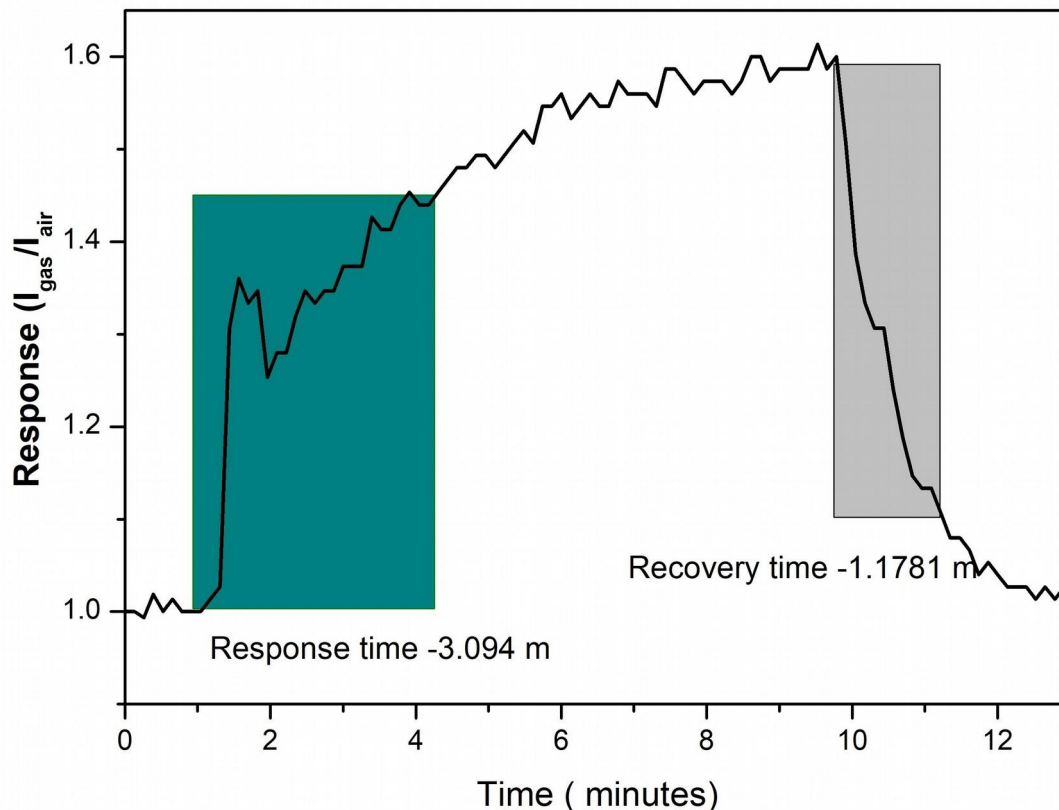
The response increases with the passage of time and eventually stabilizes. It will then revert to its initial form. The response time for the CuO is 3.094 minute and recovery time is 1.1781 minute

CHAPTER 5
CONCLUSION

The copper oxide (CuO) thin film was synthesized using the sol-gel spin coating method, and comprehensive characterization was conducted to assess its properties. The XRD data from the synthesis of nano copper oxide thin film utilizing sol-gel spin coating reveals a compelling congruence with the standard pattern, demonstrating a successful replication of copper oxide lattice constants.The process described involves synthesizing a thin film of copper oxide (CuO) using the sol-gel spin coating method. Sol-gel processes are widely used for creating thin films of various materials by dispersing nanoparticles in a solution (sol) and then allowing them to gel and form a solid material. Spin coating is a technique where a liquid is spread onto a substrate by spinning it at high speeds, resulting in a thin, uniform film

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