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Nickel Oxide Thin Film: Room Temperature Sensing Properties for Ammonia Gas

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ABSTRACT
The detection of ammonia gas is crucial in various industries due to its toxic and corrosive nature. Nickel Oxide (NiO) has emerged as a promising material for gas sensing applications owing to its unique properties. Thin films of NiO have been explored for their potential in detecting gases at various temperature. In this study, nickel oxide (NiO) thin films were deposited securely and efficiently using a convenient and affordable spin coating technique followed by annealing at 250^0c. Characterization of the structural and optical properties of NiO thin films were performed using X-ray diffraction (XRD), scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDX) and ultraviolent (UV) – visible spectroscopy. The XRD patterns revealed that all the prepared samples exhibited a cubic structure which matched well with the joint committee on powder diffraction standards (CPDS) database. The scanning electron microscopy (SEM) images revealed a homogeneous surface morphology for the films. The prepared samples exhibited high transparency, as evident from the transmittance spectra, and the absorption spectra were used to calculate the optical energy band gap. The prepared samples were then tested for gas sensing applications using a static gas sensing setup to detect various fuel gases such as ammonia at different concentrations (ppm) at room temperature. The result showed that the NiO thin films exhibited good selectivity and high sensitivity towards ammonia. The time response characteristics of the fabricated sensor, including response and recovery times, we calculated and recorded.

Keywords:
Band gap,
Characterization,
Sensing Properties,
Structural Properties,
Thin Films.

INTRODUCTION

Miniaturized gas sensors have been a major research focus over the past few decades, driven by their immense potential in addressing various industrial, health, and environmental challenges (Hung *et al.,* 2020). Various metal oxide semiconductors, including tin dioxide (SnO_2), Zinc oxide (ZnO), tungsten trioxide (WO_3), nickel oxide (NiO), copper oxide (CUO), and titanium dioxide (T_iO_2), have been widely researched for their potential applications in gas sensors. These sensors are crucial for detecting hazardous gasses, thereby enabling effective environmental monitoring (Liu and Liu; 2018). Among the above listed semiconductors, nickel oxide (NiO_2) is the most significant p-type semiconductor, characterized by a wide bandgap energy ranging from 3.6 to 4.0ev. Recent research on p-type semiconducting metal oxides has highlighted the growing interest in investigating the properties of nickel oxide thin films. This increased attention is attributed to the unique characteristics of nickel oxide, including its affordability, outstanding electrical properties, and excellent chemical stability (Yan *et al.,* 2015). The deposition of nickel oxide thin film has been achieved through multiple physical and chemical methods, including spray pyrolysis, sol-gel spin coating, chemical bath deposition, RF sputtering and electron beam evaporation (Gozukizil and Nayman, 2024).

Ammonia is a colourless gas with a pungent smell (Crivello *et al;* 2021). Exposure to ammonia at levels exceeding safe concentrations can lead to severe health issues, including skin, eye, and respiratory problems. Furthermore, ammonia can react with environmental acids to form aerosols, contributing to greenhouse gas emissions and associated environmental concerns (Arum *et al.,* 2020). Sensors with in-built sensing and alert mechanisms are designed to detect and provide early warning of hazardous gas concentrations (Bhati *et al.,* 2020).

Thin film semiconductor sensors, produced using a variety of deposition techniques, are extensively employed. The flexibility afforded by these methods allows for the creation of surfaces with specific structural and surface characteristics, catering to diverse application requirements (Chua *et al.,* 2021).

The preference for metal oxide semiconductors stems from their expedited production timelines, ease of surface integration, economic viability, and compact portability (Nechepurenko *et al.,* 2021).

Nickel oxide (NiO) has found widespread application in the field of photovoltaics (Olajire and Mohammed, 2020).

MATERIALS AND METHODS

Nickel oxide (NiO) thin film has been prepared using cost-effective sol-gel spin deposition method. In this study, a 0.1M of nickel acetate solution was prepared by dissolving the calculated amount of nickel acetate tetrahydrate in 40ml of 2-methoxyethanol, followed by stirring for 15 minutes.

A few drops of monoethanolamine were added to achieve a clear and homogeneous solution, which was then continuously stirred at 70^0C for I hour. The resulting solution was subsequently aged at room temperature for 24 hours. The aged solution was then utilized for film deposition via the sol-gel spin coating method. Prior to deposition, plain glass substrates (30mm x 25mm x 1.34mm) underwent ultrasonic cleaning for 20 minutes, using deionized water and acetone, followed by drying with nitrogen gas. Subsequently, approximately 0.2ml of nickel acetate precursor was deposited onto the pre-cleaned glass substrate and spin – coated at 3000rpm for 30 seconds.

The deposited film was annealed on a hot plate at 100^0C for 15 minutes to remove excess organic residues. This cycle of deposition and thermal treatment was repeated eight times to achieve the desired film thickness. The nickel oxide thin films, as initially prepared, underwent a heat treatment process, where they were annealed at 250^0 for a duration of 2 hours.

The structural properties of the prepared thin films were characterized using X-ray diffraction (XRD) with Cr – K∝ radiation (n=2.2897A^0) scanning 2θ values from 20^0 to 150^0. Additionally, the surface morphology and elemental composition of the films were examined using scanning electron microscopy (SEM) coupled with energy dispersive X-ray analysis (EDX).

The optical properties of the samples, including transmittance and optical band gap, were investigated using a UV – visible spectro-photometer over a wavelength range of 200 – 900nm. To characterize the gas sensing behaviour of the NiO thin film gas sensor, a bespoke gas sensing system was utilized.

RESULTS AND DISCUSSION

The XRD Pattern

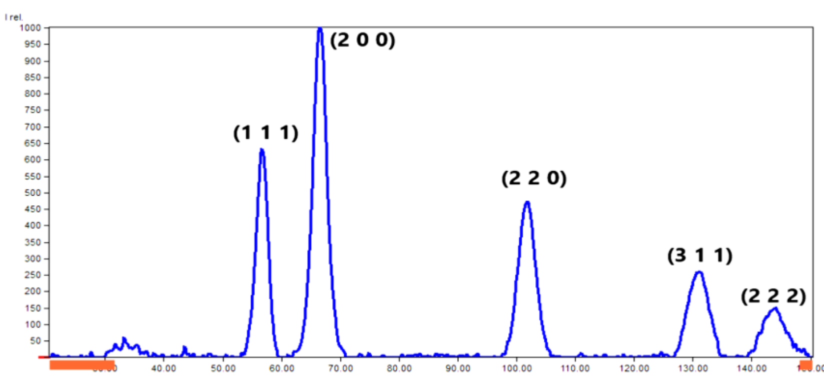

Figure 1: XRD pattern of nickel oxide thin film

The X-ray diffraction (XRD) pattern of the nickel oxide thin film deposited by the spin coating method employed in this study is as shown in figure 1 above. The XRD pattern confirms that the nickel oxide has a crystalline structure with a cubic morphology.

The diffraction pattern reveals sharp peaks corresponding to the (111), (200), (311) and (222) planes with the (200) plane exhibiting the highest intensity.

The Scanning Electron Microscopy (SEM) Nature

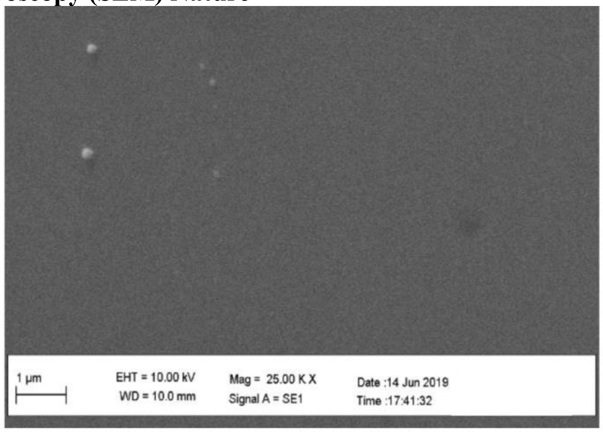

Figure 2: Surface and morphological studies of nickel oxide thin film

The scanning electron microscopy (SEM) image of the NiO thin film presented in Figure. 2 reveals a uniform surface morphology devoid of cracks.

The EDS Spectrum of the NiO Film

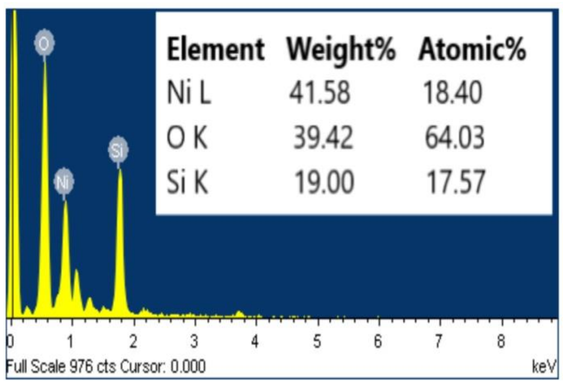

Figure 3. EDS Spectrum

Figure 3 above illustrates the EDS spectrum of the NiO thin film prepared by spin coating technique. The EDS spectrum of the NiO fim reveals the presence of nickel (Ni), oxygen (O), and silicon (Si). The spectrum shows that nickel and oxygen are the primary constituents, with weight percentages of 41.58% and 39.42%, respectively, and atomic percentages of 18.40% and 64.03% respectively. A smaller amount of silicon, likely from the substrate is also detected, accounting for 19.00% of the weight and 17.57% of the atoms.

The Optical Transmittance Spectra of NiO Thin Film

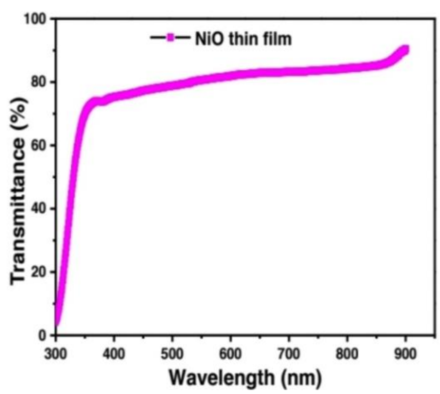

Figure 4: Optical properties of nickel oxide thin films

As shown in figure. 4, the optical transmittance spectrum of the nickel oxide thin film indicates a high degree of transparency achieving a transmittance of 90.5%.

The Optical Band Gap of NiO thin Film

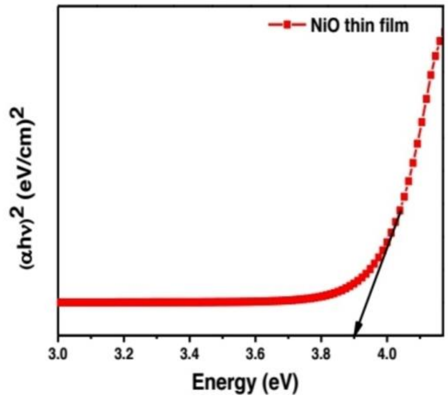

Figure 5: Tauc plot

The optical band gap of the nickel oxide (NiO) thin film was determined using the Tauc plot shown in Figure. 5, where the absorption coefficient squared is plotted against photon energy. By extrapolating the linear portion of the curve to the X-axis, the optical band gap value was obtained (Ternel *et al.,* 2018). The calculated optical band gap was found to be 3.9 eV, which is consistent with the previously reported value by Ternel *et al.* (2018).

Gas Sensing Properties

The thin film gas sensor was fabricated by attaching two silver electrodes, spaced 4 *mm* apart to the surface of the prepared NiO film, and the annealing at 100^{0}C for 1 hour using a hot plate. The sensing measurements were performed using an integrated sensing an alert system, comprising an air-sealed vacuum chamber, hot plate, thermocouple, and simple circuit.

The response of the gas sensor was calculated using the formula; Response (S) = R_g/R_a, where R_g and R_a are the electrical resistances of the fabricated sensor in the presence of analyte gas and air, respectively, as described by Nechepurenko *et al.,* (2021) for p-type semiconductor materials.

The sensor's response to various reducing gases was evaluated by exposing the sensor to targeted gases with concentration ranging from 10 ppm to 500 ppm at an operating temperature of 30^{0}C. Additionally, the selectivity, response and recovery times of the fabricated sensor were assessed.

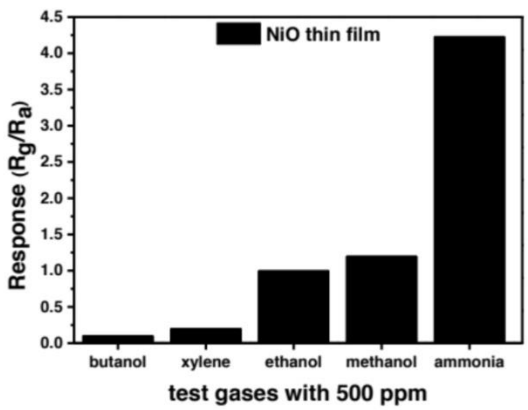

Figure 6(a): Selectivity of nickel oxide thin film gas sensor towards various test gases with concentration 500 ppm

Fig 6(a) illustrates the selectivity of the NiO sensor towards various analyte gases, including butanol, xylene, ethanol, methanol, and ammonia, each at a concentration of 500 ppm. As evident from the histogram, the sensor exhibits the highest sensitivity towards ammonia, outperforming its response to the other tested gases.

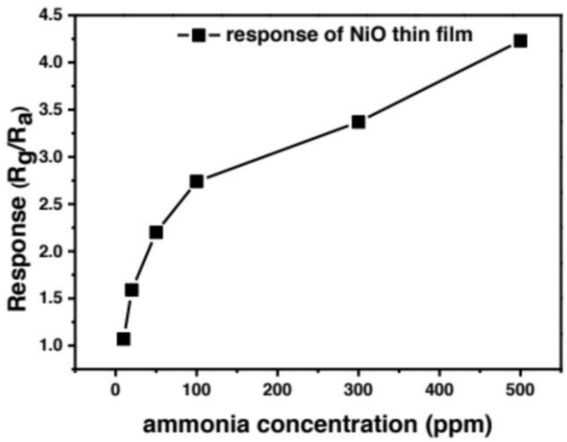

Figure 6(b): Response of sensor towards different concentrations of ammonia

Figure 6(b) illustrates the response of the fabricated NiO sensor to ammonia gas, with concentrations ranging from 10 ppm to 500 ppm. The graph reveals that the sensor's response increases proportionally with the concentration, attributed to the greater availability of analyte gas molecules on the sensor's surface at higher concentrations.

The transient response of the fabricated NiO gas sensor to 300 ppm ammonia is illustrated in figure 7.

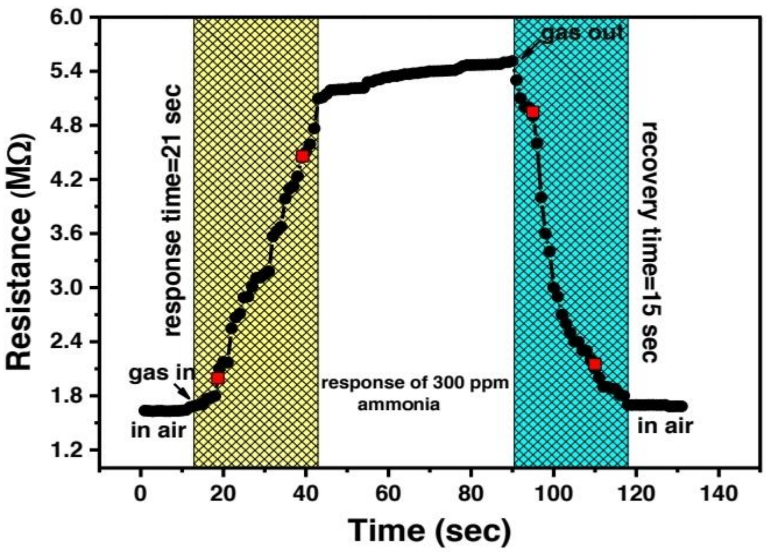

Figure 7: Transient response of the fabricated NiO gas sensor to 300 ppm ammonia

From the plot, response and recovery times were evaluated as 21 seconds and 15 seconds respectively. This implies moderate response time (the sensor takes 21 seconds to reach a stable response, which might be suitable for some applications). The result also shows faster recovery time (that is the sensor recovers relatively quickly (15 seconds), indicating good reversibility). This result compared favourably with the findings reported by Nechepuyrenko et al. (2021).

CONCLUSION

A nickel oxide thin film was successfully fabricated using spin coating, and its structural analysis confirmed the formation of cubic structured nickel oxide. The film's surface morphology revealed a uniform crack free texture. Optical studies showed the film to be highly transparent with an optical band gap of 3.9 ev. Furthermore, the nickel oxide thin film-based gas sensor demonstrated excellent selectivity towards ammonia with a response time of 21 seconds and a recovery time of 15 seconds.

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