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Physics

SYNTHESIS OF NiO THIN FILMS AND NANOPARTICLES USING A MICROWAVE-ASSISTED APPROACH FOR CO_2 GAS SENSORS

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ABSTRACT The objective of this study is to present the microwave-assisted fabrication of nickel oxide (NiO) thin films and nanoparticles for applications such as sensors for detecting carbon dioxide (CO_2) gas. The NiO was made using a microwave assisted technique utilizing 800 W of energy and different times of exposure to microwaves (30, 60 & 90 min.) to fabricate the thin films and nanoparticles. Structural and morphological properties of the NiO samples were examined by x-ray diffraction (XRD) and scanning electron microscopy (SEM). XRD examination showed that the cubic NiO phase was developed from strong peaks at 2θ angles of 36.8°, 42.9°, and 62.5° corresponding to the (111), (200), and (220) crystallographic planes. SEM examination revealed that the NiO samples contained interconnected nanoparticles and exhibited a porous microstructure. The performance of the NiO samples as sensors for CO_2 gas was examined at 300°C and it was shown that they could detect CO_2 at concentrations of 500 ppm and 1000 ppm. The advantages of the microwave-assisted synthesis process are lower processing time, less energy required for processing, and greater control over the particle morphology than traditional techniques. Therefore, the microwave-assisted synthesis process provides a suitable alternative for developing highly sensitive gas-sensing materials. Exposure to CO_2 at an optimized operating temperature of 300° C triggered a distinct increase in electrical resistance. This donor-type response deviates from classical p-type semiconductor behavior and is governed by a reversible surface acid-base interaction, wherein Lewis-acidic CO_2 coordinates with basic chemisorbed oxygen to form a temporary surface carbonate phase that suppresses majority hole carriers.

KEYWORDS :

INTRODUCTION

The importance of gas sensors for the detection of hazardous gases in a variety of environments, such as industrial, environmental, and medical, has made them a necessity in recent years. They provide important information for identifying and monitoring dangerous gas pollutants in several types of industrial, environmental and residential settings. As gas-sensing materials, metal oxides have been identified as having superior sensitivity, stability, and manufacturing ease and have consequently received a lot of attention. One of these materials is nickel oxide (NiO), a p-type semiconductor with a broad bandgap, which is considered an ideal material for CO_2 detection because of its high adsorption ability for many types of gases, good electrically conductive properties, and excellent chemical stability. Microwave-assisted synthesis of nano-structured materials like NiO offers some advantages over traditional methods, such as shorter reaction times, better crystallinity, and better control of particle size and shape. It has been shown that microwave-assisted synthesized NiO nano-particle and thin-film gas sensors have improved sensor properties, including reduced detection limits and faster response times compared to their traditionally synthesized counterparts. Various studies have investigated factors influencing the sensitivity of metal oxide gas sensors, including those based on NiO and other metal oxides [2]. As a result of the growing need for developing gas sensors that can perform at high levels for use in safety systems for security, industry, medicine and environmental protection, there is considerable interest in developing high-performance gas sensors. Currently, MOS-based chemiresistive gas sensors represent the most commonly used sensing technology in commercial applications due to their high sensitivity, relatively low price and good stability [2]. MOS-based chemiresistive gas sensors rely heavily on the structural and morphological features of the sensing materials, namely, their surface area, particle size and crystal structure, to achieve improved sensor performance [3]. A thorough study of NiO-based gas sensors and the majority of production techniques for both thin film and nanoparticle forms of NiO [4]. The enhanced gas sensing properties of NiO nano-structures obtained through microwave irradiation were reviewed in [5]. Due to its unique combination of high surface area, high sensitivity, low cost, and environmentally friendly properties, NiO nanoparticles have gained much attention for their potential application as gas sensing materials. NiO is especially suitable for selective gas detection applications due to these properties [6]. The p-type behavior of NiO is useful for gas sensing mechanisms because the interaction with the target gas results in detectable changes in electrical resistance through changes in hole concentration. Microwave-assisted synthesis of metal oxide nano-structures has been successfully employed in a multitude of systems and applications. Through controlling the nucleation and growth process of metal oxide nano-structures, it allows for a higher degree of crystallinity and morphological control of the resulting nano-structures. Additionally, microwave synthesis presents an attractive route for large scale production, primarily due to its environmentally-friendly nature corresponding to the basic principles of green chemistry. Of all metal oxide systems, NiO nano-structure is among the most frequently studied, primarily due to the superior gas sensing capabilities, high chemical stability and broad bandgap of this system. Studies utilizing microwave-assisted synthesis of NiO nano-structure demonstrate the feasibility of the utilization of these structures in gas sensors where the controlled morphology and increased surface area yield improved sensitivity and selectivity [7, 8, 9]. The fabrication of microheaters and interdigitated electrodes (IDEs) is a critical step in the development of semiconductor-based gas sensors and microsystems. Silicon wafers are widely used as substrates due to their thermal stability, mechanical robustness, and compatibility with microfabrication processes. Photolithography combined with thin-film deposition techniques enables precise pattern definition at micrometer scales. The NiO oxide thin film samples were produced by a number of different methods such as plasma-enhanced and reactive sputtering [10, 11, 12]. Due to the increasing need for accurate measurement of carbon dioxide levels in industrial, environmental and medical settings, it is essential to develop and utilize reliable CO2 sensing technologies. In particular, due to its impact on climate change and role in air quality management systems which are commonly found in homes and vehicles, CO_2 sensors are highly valuable for detecting CO_2. As a result of their high sensitivity and stable response over time, metal oxides have been extensively investigated as a method for detecting CO_2. In addition to these beneficial characteristics, NiO has also demonstrated excellent suitability for use as a p-type semiconductor with a relatively large band gap (3.6 eV to 4.0 eV) that allows it to be used for detecting CO_2 through the interaction of CO_2 with the NiO surface and the modification of the hole accumulation layer that occurs when CO_2 interacts with the NiO surface; however, the effectiveness of the CO_2 sensing ability of NiO depends upon the structural properties of the NiO. Although the automotive exhaust sensor market has achieved significant volume production of multi-sensor types (e.g., CO_2, NOx, and O2), there is currently a scarcity of reliable information regarding CO_2 sensor production. Therefore, the primary objective of this research study was to develop an optimized microwave-assisted synthesis procedure for NiO thin films and nanoparticles to determine the best process conditions for the production of NiO samples that will exhibit suitable structural and morphological properties for use as gas sensors.

Experimental Procedure

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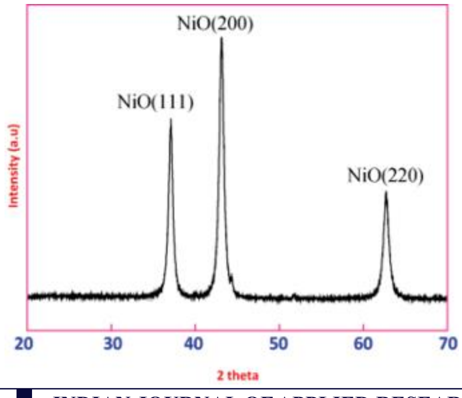
The synthesis process started by dissolving 560 mg of Nickel(II) acetylacetonate ($C_{10}H_{14}NiO_4$) into 15 ml of Ethylene Glycol ($C_2H_6O_2$), contained within a 50 ml round bottom flask. The solution was pre-heated for 60 seconds under high power microwaves to initiate metal-ligand coordination. 560 mg of sodium acetate was then added to the solution as both a nucleating and stabilizing agent. After vigorous stirring for 10 minutes, a clear and homogeneous precursor solution was obtained, confirming that the organometallic nickel source had been fully dissolved.The precursor solution was then transferred to a specially designed microwave compatible reaction vessel (working at 2.45 GHz) and heated for 30, 60, and 90 min at a constant power of 800 W. Microwave irradiation is used to heat uniformly throughout the volume of the solution, promoting the rapid nucleation and growth of the nickel based nanostructures. Once the heating cycle has completed, the system was cooled to room temperature. The resulting precipitate was collected through a high speed centrifuge. Following collection, the product underwent a series of washing steps to remove any remaining organic residue and/or unreacted precursors: Two cycles of rinsing with DI water, Single rinse with absolute ethanol and Dried until the powder reached an intermediate phase (nickel complex). Thermal annealing was performed on the intermediate products at a ramped temperature of 1°C/min from 20°C to 400°C for 2 h. A slow ramping rate is necessary to avoid collapsing the morphology of the particles while maximizing crystallinity. Once the desired temperature has been attained, the samples are left at that temperature for 2 hours to allow the oxide to form completely. The crystalline structure and phase purity of the NiO nanostructures synthesized were determined by utilizing a RIGAKU MINIFLEX II Desktop X-ray Diffractometer with Cu-Kα radiation (λ= 1.542 Å) and variable output (300W). Characterization confirmed the FCC crystallographic structure of the NiO. Platinum interdigitated electrodes (IDEs) and microheaters were fabricated on silicon substrates using standard photolithography and DC sputtering techniques. The silicon wafers were sequentially cleaned in acetone, isopropyl alcohol, and deionized water, followed by nitrogen drying and dehydration baking at 120°C for 10 min to improve photoresist adhesion. A positive photoresist (Shipley S1813) was spin-coated at 3000 rpm for 30–40 s to obtain a thickness of ~1.2–1.5 µm and soft baked at 90–100°C for 60 s. Pattern transfer was achieved by UV exposure (365 nm, 80–120 mJ/cm²) through a chrome photomask, followed by development in MF-319. Platinum (100-200 nm) with an optional titanium adhesion layer (10 nm) was deposited by DC sputtering at 3-5 mTorr in argon atmosphere. Lift-off in acetone produced well-defined platinum IDEs and microheaters suitable for microsensor applications.

Gas sensing evaluations were conducted within a custom-engineered, low-dead-volume cylindrical testing chamber equipped with an inner quartz tube liner to eliminate parasitic wall reactions. The sensor substrate was mounted inside the quartz tube and housed within an external furnace. The operational temperature was maintained at a constant 300° C via the furnace and regulated using the integrated platinum (Pt) micro heaters; the highly linear temperature coefficient of resistance (TCR) of the Pt elements ($3.92\ 10^{-3}\ K^{-1}$) ensured localized thermal stability and precise monitoring under operational thermal stress.

Dynamic gas atmospheres were generated using a calibrated gas delivery system integrated with a Netrack Gas System (ALICAT-2) mass flow controller (MFC) manifold. Dry synthetic air served as both the calibration baseline/carrier gas and the diluent matrix. A target concentration of 1000 ppm CO_2 was established by precisely metering and mixing the analyte gas with the synthetic air background under steady-state flow conditions. Real-time sensor resistance variations (R) were acquired continuously using a Keithley 237 high-voltage



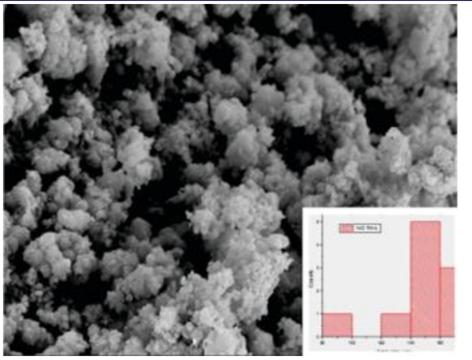


Figure 1: (a) NiO XRD Diffraction Patterns b) SEM Micrograph Source Meter.

RESULTS AND DISCUSSION

Structural and Surface Morphology

Figure (1a) identified that the peaks at the 2θ angles of 36.8º, 42.9º and 62.5º were indexed to the (111), (200), (220) planes, respectively. The X-ray diffractometer (XRD) pattern shows several distinct peaks that represent the characteristic reflections of the NiO crystal structure. It can be seen from the XRD pattern that the two most intense peaks, i.e., (200) and (111) show that the NiO phase is cubic in nature and present in the thin film sample [14]. The relative intensity and location of these peaks give an idea of the degree of preferred orientation, crystallinity and lattice parameters of the NiO material. These peaks have been found to have good agreement with JCPDS Nos. 45-1027 and 04-0850 [22, 23]. The first order diffraction peak at $2\theta = 36.8°$ represents the fundamental lattice plane of the FCC structure and is indexed to the (111) plane. The second order diffraction peak at $2\theta = 42.9°$ also represents another important lattice plane in the crystal structure and is indexed to the (200) plane. The third order diffraction peak at $2\theta = 62.5°$ represents a higher order lattice plane and is indexed to the (220) plane. This type of diffraction peaks are commonly found for nickel oxide nanoparticles and are indicative of the rock salt (NaCl-type) crystal structure of NiO. The morphology and XRD data suggest that the tips of the produced NiO nanoparticles are both spherical and cubic in shape. The time of exposure to microwave radiation affects the morphology of NiO materials.

A SEM image of NiO (nickel oxide) nanoparticles is shown in figure (1b). The network of interconnected structures and agglomeration of nano-scale particles, and their complicated and porous shapes, are clearly visible. The magnified images enable the observation of the details of the size distribution and surface properties of the nanoparticle sample. The graph on the right appears to be a histogram displaying the size distribution of NiO nanoparticles. One of the important features of the studied sample may be the significant red peak indicating that many of the particles are located around a specific size interval [15].

Gas sensing Properties

The focus of much recent research has been on developing new sensing device architectures and nanofabrication techniques as well as improving the reliability of sensor devices to meet the growing need for improved speed and sensitivity of sensors. The ratio of CO2 and dry synthetic air flow rates were adjusted to control the final gas concentration, while utilizing a dynamically controlled gas dilution system.

Gas sensors typically operate by performing three primary functions: (1) receptor function - in which the sensing material interacts with the target gas molecules; (2) transducer function - in which surface reactions cause the electrical properties of the material to be altered based upon the grain size and grain boundary; (3) utility function - representing the percentage of grains involved in the process of detecting the presence of the target gas. The sensor sensitivity is defined as the change in the electrical resistance of the sensing material to the presence of the target gas and the measured environmental parameter [16]. In this paper, NiO nanoparticles were prepared using a microwave assisted synthesis technique, followed by processing the particles into thin film structures suitable for gas sensing. The morphology of the synthesized NiO nanoparticles was characterized as flower-like aggregates via both SEM and XRD analysis, resulting in large surface areas providing enhanced opportunities for gas-surface interactions. Additionally, these NiO nanoparticle thin films were successfully incorporated into sensor structures and demonstrated potential as suitable materials for use in CO_2 gas sensing applications.

Delivery and Specifications for CO_2 Gas: A 1000 ppm CO_2 level has been measured by an ALICAT-2 MFC in a dynamic dilution gas system that provides extremely precise control of CO_2 concentrations. A micro-heater was incorporated into the substrate below the NiO sensing layer to heat the sensor device. The micro-heater was powered by electricity and heated the sensing area uniformly, thereby allowing the sensor to operate at temperatures ranging from 300–350 °C. The operating temperature of the sensor was controlled through regulation of the amount of electrical power delivered to the micro-heater via a stable DC power source. The temperature was monitored via a calibrated temperature sensor and measurements were made after the temperature had reached thermal stability to provide consistent and reliable resistance changes. The final gas concentration was determined as the ratio of the CO_2 and air flow rates.

$$C_{final} = C_{source} \times \frac{f_{CO_2}}{f_{CO_2} + f_{air}}$$

The sensor was conditioned prior to each measurement with dry synthetic air until a stable resistance (Ra) could be measured. After conditioning the sensor was then exposed to a CO_2 concentration of 1000 ppm and the changes in resistance were monitored until a stable resistance value (Rg) could be measured. The production of CO_2 sensors has become massive in scale. Every single year tens of millions of CO_2 sensors are made. The baseline resistance of a p-type semiconductor such as NiO is determined by the concentration of holes in the surface hole accumulation layer. When exposed to CO_2 gas molecules, the concentration of holes on the surface is decreased due to electron compensation from surface reactions, which increases the resistance of the sensor. The NiO thin film used in this application is highly sensitive to CO_2 gas because of its p-type semiconductor characteristics at operating temperatures near 300-350°C [17]. The p-type semiconductor characteristics of NiO thin films provide a high sensitivity to CO_2 gas detection [18]. Surface reaction and gas diffusion are two very important processes that determine the gas sensing capability of materials based on oxides. Generally, the gas sensing capability of materials based on oxides may be determined by the surface reaction and gas diffusion mechanisms [19, 20]. The operation of the device relies upon measuring changes in electrical resistance of the semiconductor surface in response to interaction with gas molecules.

The anomalous electrical response of the p-type NiO sensor under CO_2 exposure—characterized by a distinct increase in electrical resistance—deviates from classical oxidizing gas models and is attributed to a reversible surface acid-base reaction. At an operating temperature of 300 °C , the Lewis-acidic CO_2 molecules interact with the basic chemisorbed oxygen species ($O^-_{(ads)}$) on the NiO surface to form a metastable nickel carbonate ($NiCO_3$) intermediate phase via the following pathway:

$$CO_{2(gas)} + O^-_{(ads)} + h^+ \rightleftharpoons CO^{2}_{3(ads)}$$

This localized coordination effectively traps mobile holes ($h^·$) from the valence band or conversely injects electrons into the p-type core, neutralizing the majority charge carriers. The resulting depletion of surface hole density broadens the electrical charge transport barrier, driving the macroscopically observed increase in sensor resistance."

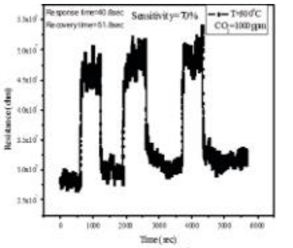


Fig. 2 (a) CO_2 gas operating at 300° and concentration 1000ppm

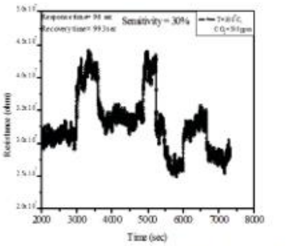


Fig. 2(b) CO2 gas operating at 300° and concentration 500ppm

4.0 CONCLUSION

The microwave-based synthesis method is being used to prepare NiO thin film and nanoparticle materials that exhibit appropriate structural and surface morphological properties for application in gas sensors. X-ray diffraction analysis confirmed that the produced NiO was single phase cubic, while scanning electron microscopy (SEM) images of the synthesized products revealed a complex network of interconnected NiO nanoparticles that exhibited high porosity. Sensor responses based on CO_2 detection were obtained at an operating temperature of 300°C; additionally, these devices demonstrated reliable CO_2 concentration sensing capabilities to both 500 ppm and 1000 ppm. The results from this study demonstrate the feasibility of utilizing microwave-assisted synthesis methods to shorten process times, reduce the total energy required during synthesis, and provide greater control over the final product's physical/chemical properties. The mechanistic investigations revealed that the sensor's response to CO_2 is driven by a reversible surface acid-base reaction rather than conventional electronic charge transfer. At 300°C, the interaction between Lewis-acidic CO_2 and basic surface oxygen species forms a meta stable carbonate intermediate. This process successfully traps mobile holes, expanding the electrical transport barrier to produce the observed macroscopic resistance increase-offering a robust framework for designing high-temperature p-type metal oxide chemi-resistors.

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