

RESEARCH PAPER

## Enhanced Structural and NO<sub>2</sub> Gas Sensing Properties at Different Temperatures of Zinc Oxide (ZnO) Nanoparticles Prepared by DC Plasma

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### ABSTRACT

The aim of this study to prepare and develop zinc oxide (ZnO) nanostructures using DC-Ar plasma treatment. X-ray diffraction (XRD) results confirmed that DC-Ar plasma treatment preserved the pure hexagonal phase of zinc oxide at the main crystalline levels without the formation of any secondary phases. Ionic bombardment with active argon ions improved the degree of crystallinity and atomic rearrangement, generating additional oxygen holes. The sensor exhibited typical n-type semiconductor behavior; the reaction with NO<sub>2</sub> gas increased the surface depletion layer thickness, thus significantly raising the electrical resistance. We found that the sensor's performance was highly temperature-dependent, with the highest sensitivity (93.66%) recorded at the optimum temperature of 200 °C, compared to 53.98% at room temperature, due to the activation of chemically adsorbed oxygen ions (O<sup>2-</sup>). The results demonstrated that DC-Ar plasma technology for modifying zinc oxide nanosurfaces is effective in fabricating nanomaterials and manufacturing highly efficient and sensitive environmental gas sensors for detecting NO<sub>2</sub> gas at different temperatures.

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### INTRODUCTION

One of the most hazardous gaseous pollutants from combustion processes in transportation and various industries is nitrogen dioxide (NO). It is associated with respiratory diseases, lung infections and air quality degradation [1]. NO<sub>2</sub> also participates in the formation of smog and acid rain, therefore it is a gas of great interest in current environmental monitoring systems [2]. Nanomaterials have been considered as

a promising option in the development of gas sensors in recent years due to their large surface area and high percentage of active surface atoms which enhances the surface adsorption reactions and improves the sensor's sensitivity to target gases [3]. One of the most utilized materials in this area are semiconducting metal oxides due to their chemical stability and electronic properties adequate for gas sensing processes [4]. Recently, modern techniques for the preparation of

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nanomaterials have attracted increasing attention to cold plasma technology, which can produce nanoparticles with different structural and surface properties at relatively low temperatures compared to conventional methods [5]. The cold plasma is characterized by the presence of high-energy electrons, ions and active free radicals which contribute to the enhancement of the synthesis and modification of nano surfaces leading to the improvement of the physical and chemical properties of the prepared materials [6].

The plasma treatment of nanomaterials has been shown to increase the concentration of oxygen holes and surface defects, which are important factors for improving the gas sensing mechanism by increasing active adsorption sites and increasing the transfer of electrical charges on the surface of the sensitive material [7]. Plasma prepared or plasma treated nanomaterials have also proved to have superior performance in detection of oxidizing gases such as nitrogen dioxide [8]. Temperature is considered Operating conditions are one of the most important factors affecting the performance of semiconductor metal oxide gas sensors. They control the nature of the oxygen species adsorbed on the surface and the kinetics of the interactions between the target gas and the sensor material [9]. Therefore, studying the response of the sensor to NO<sub>2</sub> gas at different operating temperatures is a crucial step in determining the optimal conditions that achieve the highest sensitivity and best response and recovery times. The present work aims to prepare zinc oxide nanoparticles by using plasma technology and to study the performance of zinc oxide nanoparticles as a sensor for nitrogen dioxide gas at different operating temperatures. The results showed that plasma technology has successfully produced zinc oxide nanoparticles that are good sensors for nitrogen dioxide gas at different temperatures. The sensing rate was enhanced with the increase of temperature.

Zinc oxide (ZnO) is one of the most extensively used oxide semiconductors for electronic and sensing applications due to its excellent physical and chemical properties. It is an n-type semiconductor with a wide direct energy gap of about 3.37 eV and a high exciton binding energy of about 60 eV at room temperature, which is suitable for numerous optical and electronic applications [10].

Due to their ease of synthesis in a variety of

morphologies, including nanoparticles, nanowires, nanorods, and nanosheets, ZnO nanoparticles have attracted a lot of attention recently. Compared to many other semiconductors, they also have a large surface area, superior chemical stability, and cheap production costs [11]. It is one of the most promising materials for creating gas sensors because of these characteristics, which improve surface interactions between the substance and ambient gases. Zinc oxide's gas sensing method depends on interactions between gas molecules and oxygen species that have been adsorbed on the material's surface. An electron-depletion layer forms and the material's electrical resistance rises when ZnO is exposed to air because oxygen molecules are adsorbed onto its surface and take electrons from the conduction band. Oxidation-reduction reactions take place when the surface is exposed to various gases, changing the concentration of free electrons and, as a result, the electrical resistance of the sensor [12]. Because nitrogen dioxide (NO<sub>2</sub>) is a potent oxidizing gas, its reaction with the ZnO surface causes more electrons to be removed from the material, increasing the thickness of the depletion layer and producing a discernible change in electrical properties that can be used for gas detection. The effective surface area, crystal size, and concentration of surface defects and oxygen holes—active locations for gas molecule adsorption and interaction—all have a major impact on the sensor's sensitivity [8]. Reducing ZnO crystals to the nanoscale greatly increases sensor sensitivity and shortens response and recovery times, as numerous studies have shown. This is explained by a higher ratio of surface area to volume and more active places for interaction. Additionally, treating ZnO using cutting-edge methods like metallic doping or cold plasma can raise the density of oxygen gaps and surface flaws, improving gas sensing capability, especially for NO<sub>2</sub> [8]. Because of these remarkable qualities, zinc oxide nanoparticles have grown in popularity as materials for creating extremely sensitive and affordable gas sensors, which makes them attractive options for industrial safety systems, environmental monitoring, and early gaseous pollution detection.

In Fig. 1 gas supply unit includes a cylinder of high-purity argon (Ar) gas, connected to a precision flow rate control system. This system includes a control panel and a calibrated flowmeter to

maintain a constant flow rate of 3 L/min through the main channel.

A high-voltage DC power supply provides an operating voltage of approximately 11 kV. The positive electrode (anode) of the power supply is connected to a 1 mm diameter metal needle that acts as a plasma ejector, while the negative electrode (cathode) is connected to a 2 cm conductive metal strip close in the aqueous solution. The gas flow is directed vertically through the needle (anode), which is positioned at a constant vertical distance of 1 cm above the surface of the liquid being treated. The electrical circuit is completed via the interconnection between the three phases (gas, liquid, and power source). The system includes an adjustable dynamic sample holder designed to support, change, and precisely align different phases under the plasma jet.

## MATERIALS AND METHODS

### Preparation of Zinc Oxide Nanoparticles

Commercial zinc powders containing impurities and a stainless-steel electrode were used. A DC plasma was generated using argon gas at a voltage of 11 kV and a gas flow rate of 3 liters per second. The thermal plasma arc erupted directly into the atmosphere for an exposure time of approximately 50 seconds. The zinc powders were fed into the plasma flame via the argon gas carrier. The temperature of the zinc powder rose instantly and rapidly, transforming the solid zinc

into a dense, rising vapor. Upon rising and mixing with the oxygen in the surrounding atmosphere, a spontaneous oxidation reaction occurred. This reaction manifested as a very dense white smoke composed of zinc oxide nanoparticles. The resulting product was then subjected to evaporation, oxidation, and cooling.

## RESULTS AND DISCUSSION

### Morphological characteristics (SEM)

Fig. 2 demonstrate the typical SEM images of ZnO. The shape of ZnO NPs is mostly –spherical continuous, full-bodied structure made up of geometrically defined, closely spaced crystalline grains. These grains are equally and regularly dispersed throughout the whole surface, with little fluctuation in size, and they take on a variety of shapes. The material’s high crystallinity, which perfectly matches the sharp diffraction peaks found from X-ray diffraction of zinc oxide nanoparticles prepared or processed using surface energy-controlled techniques like plasma technology, is responsible for the distinct crystalline surfaces and sharp edges of the pyramidal grains. In terms of gas sensing, this structure contributes to improving nitrogen dioxide (NO<sub>2</sub>) sensing efficiency. For the chemisorption of gas molecules and oxygen species, the exposed crystalline surfaces and random granular packing offer a large specific surface area and several active sites. Additionally, a dense network of grain boundaries is produced by

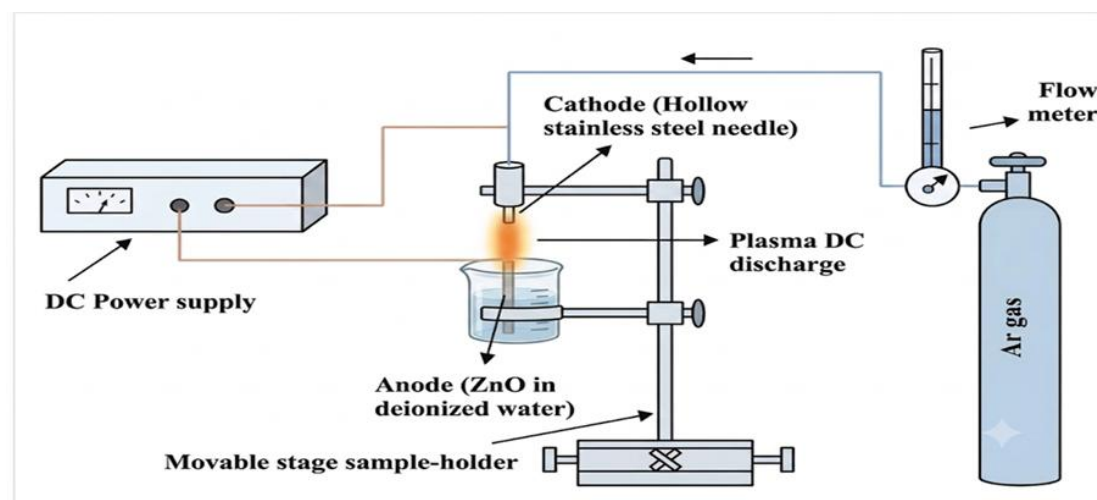


Fig. 1. Schematic of DC plasma.

the material's granular structure. When the sensor is exposed to the gas, free electrons from these zinc oxide grains are taken up by gas molecules.

#### XRD ZnO

The XRD results for the sample treated with DC plasma using argon gas in Fig. 3 showed diffraction

peaks at  $2\theta \approx 31.8^\circ, 34.4^\circ, 36.3^\circ, 47.6^\circ, 56.6^\circ,$  and  $62.9^\circ$ , corresponding to the (100), (002), (101), (102), (110), and (103) crystalline planes of the wurtzite hexagonal structure of zinc oxide. The results showed no secondary phases, indicating that the material retained its crystalline phase after plasma treatment.

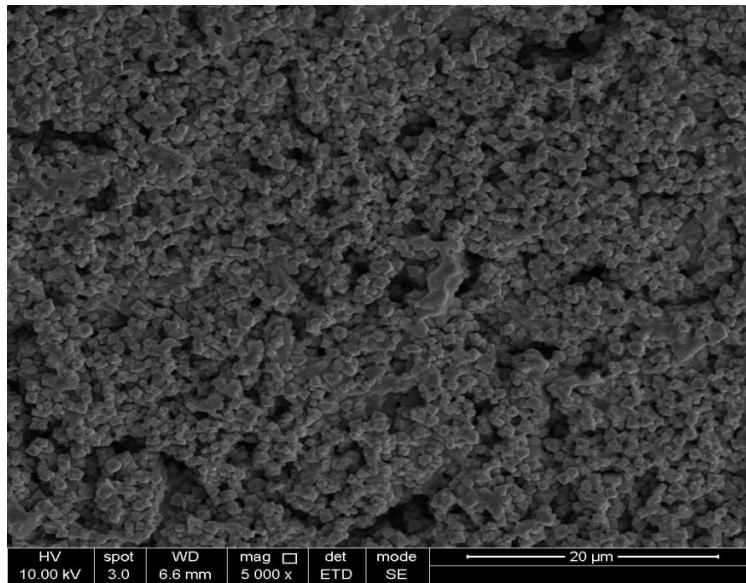


Fig. 2. SEM of ZnO.

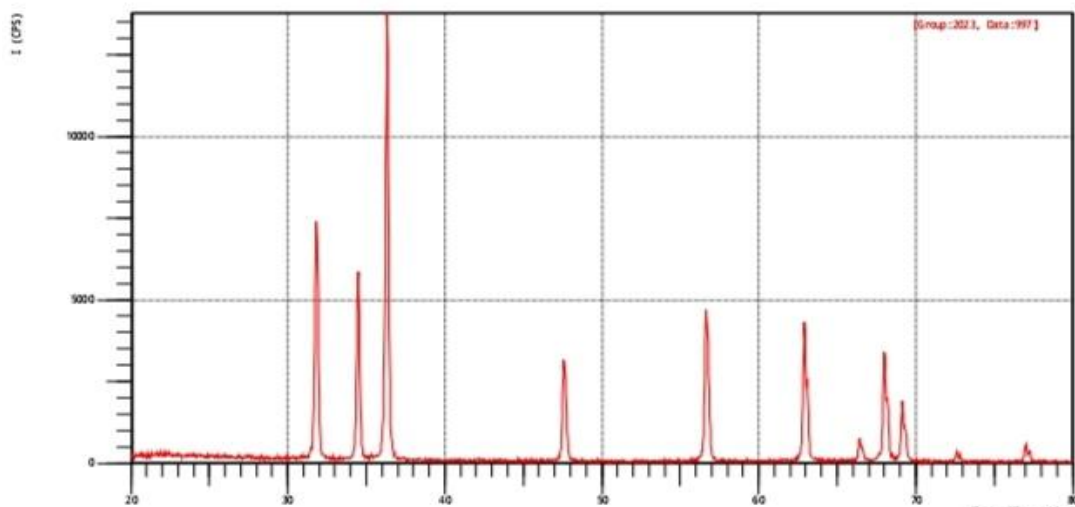


Fig. 3. X-Ray of ZnO.

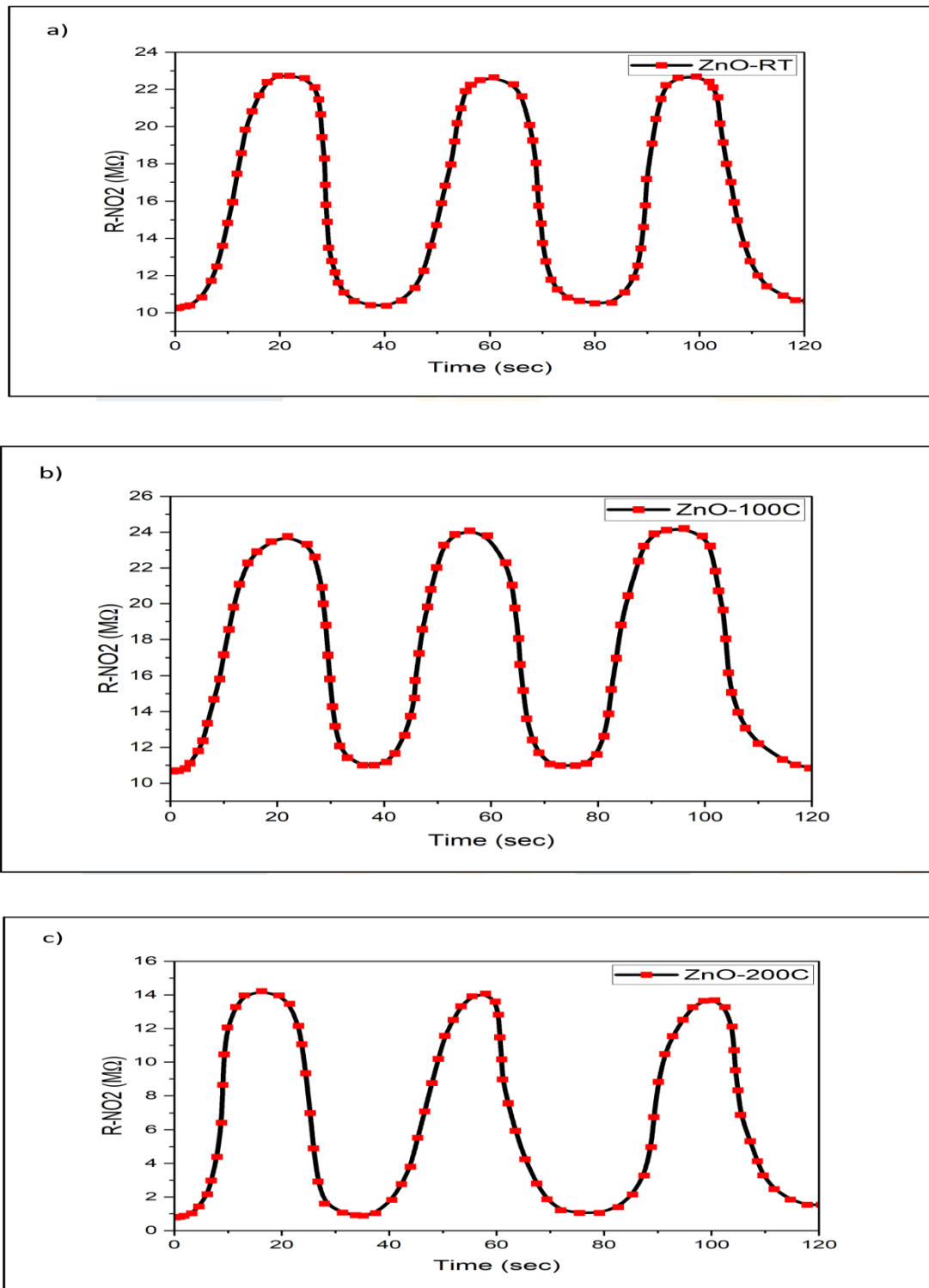


Fig. 4. Variations of resistance vs. time of ZnO film sensor upon exposure to NO<sub>2</sub> gas at operating temperatures a) RT, b) 100 °C and c) 200 °C.

Treating ZnO with argon plasma increased the crystalline phase percentage and improved crystallinity, along with higher diffraction peak intensity, without altering the material's fundamental hexagonal structure. The study also demonstrated a significant increase in the degree of crystallinity due to the interaction between the plasma's active ions and the material's surface.

This study showed that argon plasma leads to the generation of additional oxygen holes and improves the structural and electronic properties of the material while preserving the original crystalline phase. The ion bombardment of the ZnO surface by energetic argon ions, which leads to atomic rearrangement and the reduction of some structural defects.

The slight shift in peak positions and the improvement in crystallinity are attributed to changes in the concentration of intrinsic defects, including oxygen holes, without causing the collapse of the crystal lattice or the formation of secondary phases. Increasing the concentration of oxygen holes is associated with an improvement in some of the material's electronic and optical properties.

Therefore, it can be concluded that treatment with DC-Ar plasma improved the crystalline structure of ZnO and preserved the pure hexagonal phase, which is consistent with the findings of previous published studies [13].

#### Gas Sensor Measurements

##### Variation in an electric resistance function from time to NO<sub>2</sub> gas

The dynamic response curves of ZnO sensor to NO<sub>2</sub> gas at different operating temperatures, room temperature, 100 °C and 200 °C are presented in Fig. 4 and the sensitivity values (%S) and the corresponding response and recovery times are summarized in Table 1. The electrical resistance

of all curves is increased when the sensor comes in contact with NO<sub>2</sub> gas, which is a very expected behavior of an n-type ZnO semiconductor. This is because NO<sub>2</sub> gas is an electron collector that decreases the concentration of free electrons and increases the electrical resistance by increasing the thickness of the depletion layer, on the surface of ZnO [14,15].

As observed from Table 1 the sensitivity was found to be 53.98% at room temperature, 54.39% at 100 °C and later on there was a great increase in the sensitivity which was 93.66% at 200 °C. It is believed that the increase in improvement is due to the activation of the surface process, since at higher temperature the amount of chemically adsorbed oxygen species (O<sup>-</sup>) is larger, and thus interaction between NO<sub>2</sub> molecules and the surface of the ZnO increases, and the change in the resistance increases [16,17]. In addition, the response time was reduced from 18s at room temperature to 14.49s at 200 °C, suggesting that the velocity of charge transfer was improved and the process of the adsorption of the gas was accelerated on the surface [18].

In addition, the curves in Fig. 3 demonstrate good stability and high repeatability over the three successive cycles of operation, as the response has an amplitude of almost the same value without significant degradation. It is a key parameter which shows reliability and stability of the sensor's operation [19]. This slight increase in the recovery time at 200 °C (16.2 seconds) as compared to 100 °C (13.77 seconds) may be explained by the fact that the NO<sub>2</sub> molecules bind strongly to the surface-active sites at higher temperature and, hence, longer time is required for complete desorption [20]. In summary, the results indicate that the optimum operating temperature of the ZnO sensor to NO<sub>2</sub> is 200 °C, which has the highest sensitivity with a quick response and good

Table 1. Sensitivity, response and recovery times of ZnO film sensor upon exposure to NO<sub>2</sub> gas at different operating temperatures.

Sample	T (°C)	S%	tres (sec)	trec (sec)
ZnO	RT	53.98	18	14.85
	100	54.39	17.1	13.77
	200	93.66	14.49	16.2

stability. This is in alignment with the several published studies of ZnO nanosensors to detect oxidizing gases [21-23].

## CONCLUSION

the current study successfully prepared zinc oxide (ZnO) nanostructures by using argon gas DC-Ar plasma treatment. X-ray diffraction (XRD) results confirmed that the plasma treatment preserved the pure hexagonal phase of the zinc oxide at the main crystalline levels without the formation of any secondary phases. Ionic bombardment with active argon ions improved the degree of crystallinity and generated additional oxygen holes due to atomic rearrangement. The material exhibited typical n-type semiconductor behavior, where the reaction with oxidizing NO<sub>2</sub> gas increased the surface depletion layer thickness and thus raised the electrical resistance. This performance was found to be temperature-dependent; the highest sensitivity was 93.66% at the optimum temperature of 200 °C compared to 53.98% at room temperature. The sensor demonstrated excellent stability in sensing capacitance, ultimately proving that integrating DC-Ar plasma technology represents an effective and reliable strategy for defect engineering. Crystallography and the development of a new generation of highly efficient environmental gas sensors for detecting NO<sub>2</sub> gas.

## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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