# Preparation and Characterization of Indium Oxide Nanoparticles by Sol-Gel Method and its $NO_2$ Gas Sensing Properties

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## ABSTRACT

In this work, $In_2O_3$ nanoparticles were prepared by sol-gel method and deposited on quartz substrate by dip coating technique and annealed in air at different temperatures of (450, ,550 and 650)˚ C at constant time (60 min). X-ray analysis has confirmed the formation of polycrystalline cubic phase that increases in crystaline size with increasing annealing temperature. The optical properties of $In_2O_3$ nanostructure thin film were studied. The transmittance was measured in the wavelength range from (300nm to 1100 nm) for all the films. The sensitivity toward $NO_2$ gas has been measured, where $In_2O_3$ annealed at different temperatures.

## INTRODUCTION

Metal oxide nanoparticles such as indium oxide ($In_2O_3$) have unique characteristics such as good conductivity, high optical transmittance over the visible wavelength region, excellent adhesion to substrates and chemical stability and photochemical properties[1]. Indium oxide is a wide band gap n-type semiconductor with direct band gaps of 3.75 eV. Indium oxide has a cubic bixebyte structure with a lattice parameter of 10.117A˚. Also, indium oxide is an important and distinguished transparent conducting oxide (TCO)[2].

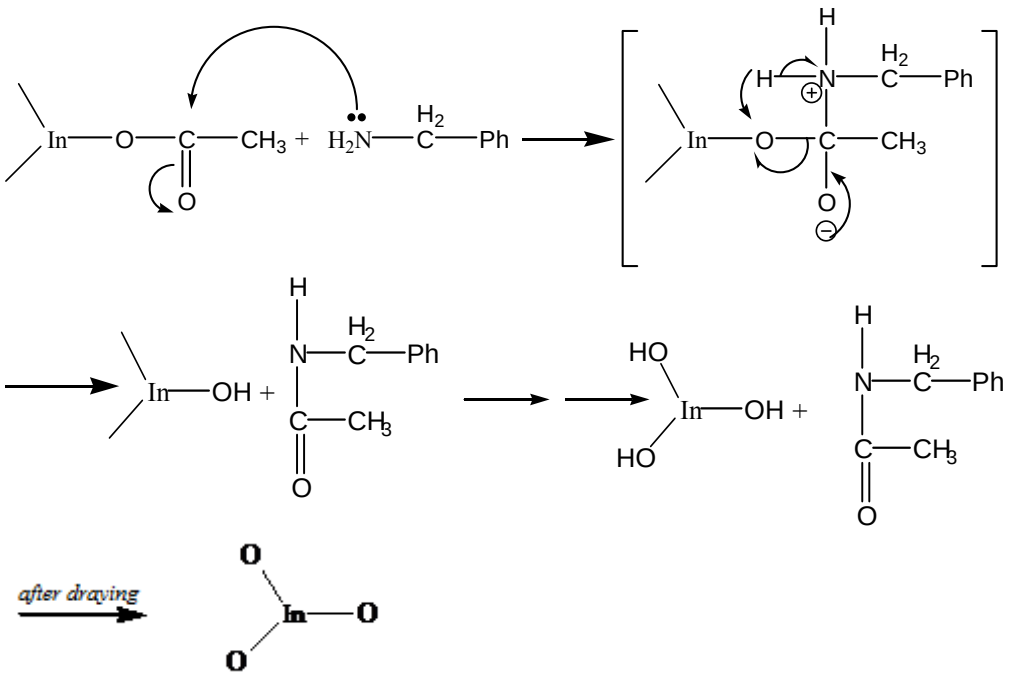
Gas sensors play an important role in detecting, monitoring and controlling the presence of dangerous and poisonous gases in the atmosphere at very low concentrations [3,4,5]. Nanostructured semiconductor gas sensors are highly sensitive and dependable, and have a performance/price ratio as good as to that of microelectronic components [6,7,8]. It is well known that the physicochemical properties which control gas adsorption on the surface of a semiconductor can significantly influence its electrical conductivity [9,10].

### Experimental procedure

### Preparation of Indium Oxide Nanoparticles.

Indium oxide nanoparticles were synthesized through the aminolysis reaction of indium acetate in the presence of benzylamine in bottom round flask.

We suggest that the substitution reaction took place between the acetate group of $In(CH_3COO)_3$ and the $-NH_2$ group of benzylamine, forming $In(OH)_3$. After reaction, white $In_2O_3$ powder was separated through centrifugation, rinsed in ethanol, and dried in a vacuum oven at 90 °C for 12 h.. The suggested aminolysis mechanism is shown in scheme (1).




**Scheme (1): Suggested mechanism of reaction between indium acetate and benzylamine**

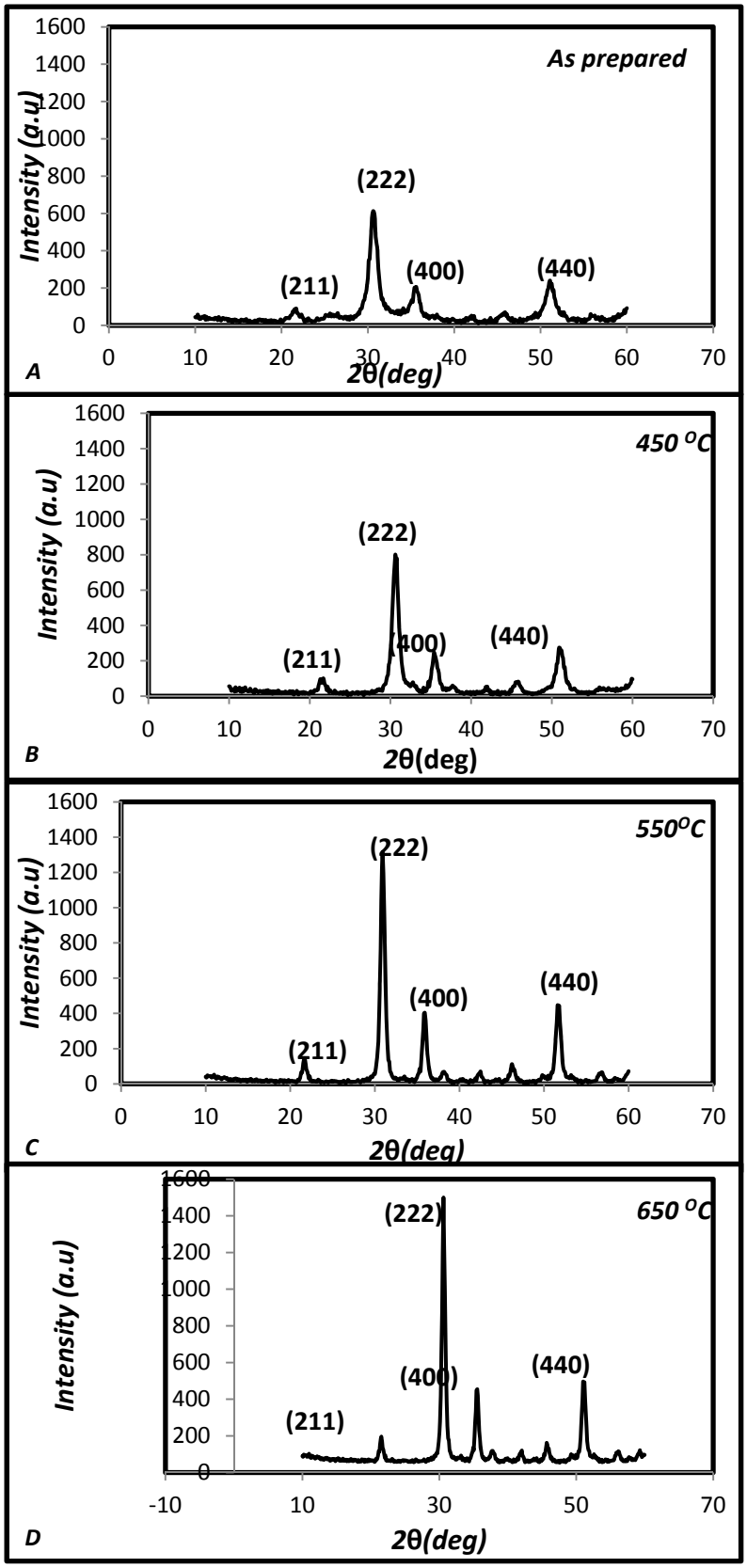
**Thin Film deposition**

The $In_2O_3$ thin film was deposited by dip coating technique. Quartz substrates were dipped in the prepared solution and withdrawn at a rate of (1 cm $sec^{-1}$ ). The substrate stayed in the sol for 60 s, and was subsequently dried for 5 min at 100°C. The number of dipping was between 8 and 15, the thickness was 200nm.

**Results and discussion**

**X-Ray Diffraction (XRD)**

The annealing temperature plays an important role in determining the structure of $In_2O_3$ nanoparticles, the X-ray diffraction patterns of indium oxide nanoparticles show high diffraction peaks showing good crystallinity. The diffraction peaks agree with those given in JCPD data card of bulk cubic $In_2O_3$ reflections from (2 1 1), (2 2 2), (4 0 0) and (4 4 0) planes, it can be seen that all the annealed $In_2O_3$ nanoparticles are polycrystalline with a cubic structure. The diffraction pattern peaks of $In_2O_3$ for all samples located at (211),(222),(400) and (440). From figure (1), it can be seen that there is an increasing in intensity of (222) orientation, this may be due to heat treatment that enhances the mobility of atoms in rearrangement processes inside the lattice. The heat energy that provided to the atoms could decrease the defect in the $In_2O_3$ nanoparticles and improve quality. This leads to decreases in Full Width at Half Maximums (FWHM) of the reflection peaks which become narrower as the particle size increases, which is a general size-dependent phenomenon within nanoparticles [11,12].




**Figure(1):XRD patterns of $In_2O_3$ nanoparticles at different annealing temperatures: (A) as-prepeared $In_2O_3$ powder (B)450 $^{O}$C (C)550 $^{O}$(D)650$^{O}$C.**

**Film Morphology**

**Surface morphology Atomic Force Macroscopic (AFM)**

The surface morphology of $In_2O_3$ nanoparticles was analyzed using atomic force microscope. Figure (2) shows a typical three and two dimensional AFM image of $In_2O_3$ nanoparticles with and without annealing at 550°C. The average grain size found to be (70-96 nm). AFM results show that the grain size increase by increasing temperature this is due to improving the crystalline of the particles [12,13,14].

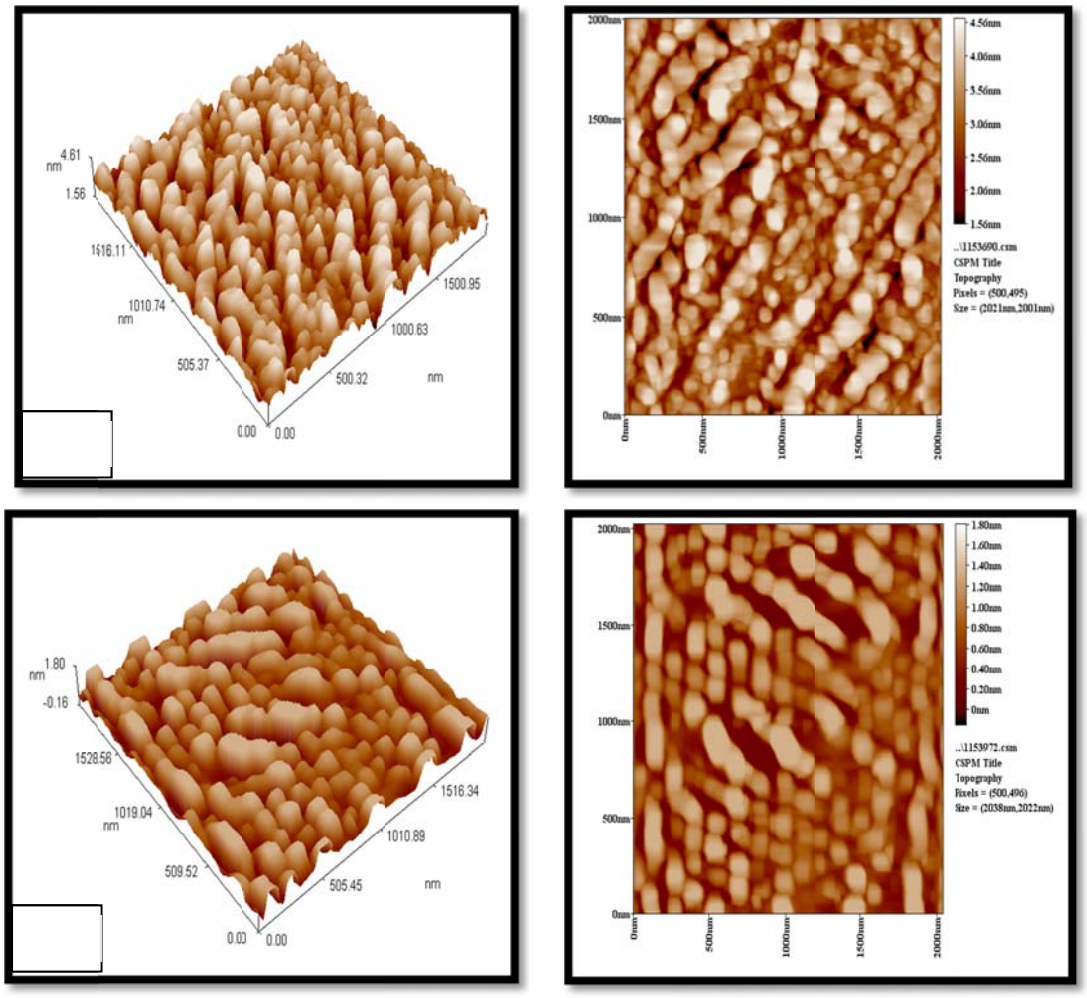



**Figure (2): (A) 3-D and 2-D AFM image for $In_2O_3$ without annealing ,(B) 3-D and 2-D AFM image for $In_2O_3$ annealed 550°C for 60 min.**

**Transmission**

The effect of the annealing temperatures (450,550,650) °C for (60 min) on $In_2O_3$ thin films is shown in figure (3). It is found that the films have high transmission at long wavelengths reach to (90%) in the visible region. The optical transmission of $In_2O_3$ thin films found to be decreases with increasing annealing temperature, this is due to increasing in carrier concentration lead to increase absorption (the higher electron concentration, the lower IR transitions) also changing in oxygen contain, also improving the crystallinity leads to increase the surface scattering of the light, the surface roughness attributed to densification and agglomeration of the crystallites at

the highest temperatures. The decreases in the UV region (below 350 nm) is due to the fundamental absorption of light [11,12].

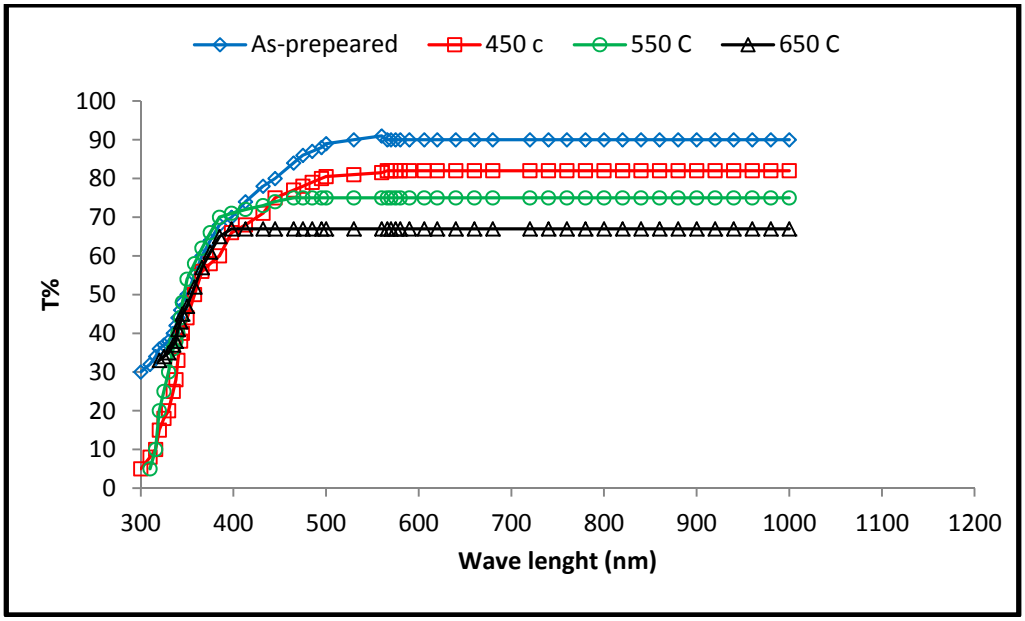



**Figure (3): UV-VIS transmittance spectra of $In_2O_3$ thin films at different annealing temperature (450°C, 550°C, and 650°C).**

**Sensing Properties**

The sensing properties for $NO_2$ were studied as a function of operating time by pure $In_2O_3$ thin film. The concentration of Nitrogen dioxide gas was (5ppm).

The gas sensitivity of $In_2O_3$ films is calculated from measuring the resistance change for thin films in air and in gas. The change in surface resistance in the presence of gas with time is measured by using equation $S=\frac{(Rg - Ra)}{Ra} * 100$ **%**

Where: Ra and Rg are the resistance of film in air and in gas respectively. Figure (4) shows the gas sensitivity of $In_2O_3$ as a function to operating time. It can be seen that the sensitivity is found to be maximum when the annealing temperature was 550°C. The annealing in air renders more oxygen vacancy generation, which enhances the gas sensitivity. The sensing materials have to be annealed at various temperatures to achieve crystallization and structural evolution. A sufficient degree of crystallinity is required to attain the desired electronic properties necessary for gas sensor application. It is observed that the sensitivity increases from 100°C to 550°C and then decreases with the further increase in the annealing temperatures [15]. It is shown that the maximum sensitivity of ( 65, 70, 80 and 74%) to $NO_2$ at annealing temperatures 100°C, 450°c ,550 °C and 650°C respectively.

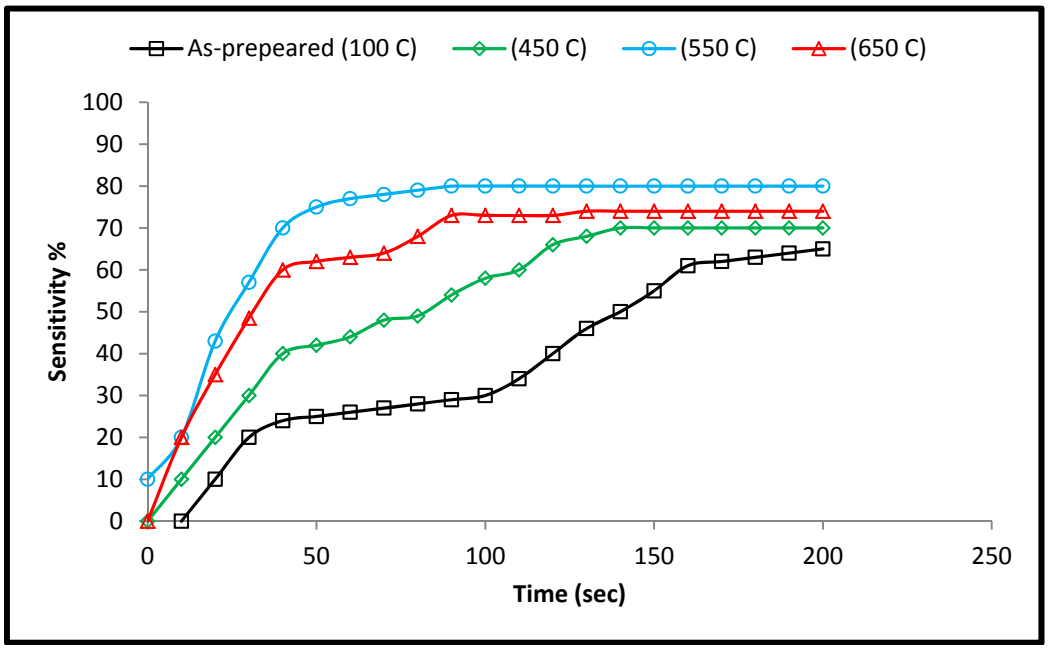



**Figure (4) :Sensitivity of $In_2O_3$ films at different annealing temperature for $NO_2$ gas.**

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