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Calcination Temperature Dependent of Hydrothermal Indium Oxide Nanostructures

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ABSTRACT

In this work, indium oxide (In_2O_3) nanostructures were successfully synthesis by hydrothermal method at a reaction temperature 180°C for 5 hour and different calcined temperatures ($300, 400$ and 500°C). The influence of the temperature on the structural and optical properties was study. The morphology and structural properties of as prepared samples were characterized by X-ray diffraction (XRD) and field emission scanning electron microscopy (FE-SEM). The structural analysis shows a highly crystalline of indium oxide with a cubic phase and the average grain size is (16.11 nm). The optical properties of In_2O_3 nanostructures were studied by using UV-visible Spectrophotometer. The transmittance of the samples prepared at different temperatures have been recorded in the wavelength rang (300-800)nm.

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INTRODUCTION

During the last few years, many researchers have been obsessed with investigating low-dimensional nanostructures due to their exceptional characteristics and applications (Andreas, 2012),(Ghim, 2011). Thanks to large surface-area-to-volume ratios and super-dense oxygen vacancies, plenty of semiconductor nanostructures, such as SnO_2 , ZnO , Ga_2O_3 , In_2O_3 and so on , have been widely applied in many fields (Niranjan *et al.*, 2013). For instance, the SnO_2 and ZnO nanowires can be used for detecting toxic gas and harmful light sensors (Jia *et al.*, 2006). In terms of optoelectronic properties, the In_2O_3 nanostructures are second to none in all nano-materials. Indium oxide (In_2O_3) is an n-type wide direct band gap (3.6 eV at room temperature) transparent semiconductor (Xiang *et al.*, 2003), (Guodong, 2011). To improve In_2O_3 properties, researchers have successfully synthesized various nanostructures, e.g., nanorods, nanotowers, nanowires and nanotubes (Young-Sik and Young-Duk, 2010). According to these literatures, it is clear that different morphologies and phase structures of In_2O_3 nanostructures lead to diverse properties. Indium oxide is an important and well known transparent conducting oxide (TCO) (Zhuangdong *et al.*, 2013). Over the past decades, many studies have been done on preparation of indium oxide (IO) and indium tin oxide (ITO) in bulk or thin film form, which are of the most important technical materials for various applications such as solar cells, sensor modules, and transparent electrode materials for optoelectronic devices (Tula *et al.*, 2009). There is a variety of synthesis techniques (routes) for preparation of indium oxide nanoparticles such as sol-gel, pulsed laser deposition and chemical vapor deposition (Sun-Jung *et al.*, 2011). Among these methods hydro-thermal processes have been widely used due to their simplicity, lower cost, and ability to control the particle size and shape (Yawei *et al.*, 2013). In this paper, we report preparation and study of the properties of indium oxide nanoparticles synthesis by hydrothermal method.

MATERIAL AND MATHOD

The indium oxide nanostructures were synthesized by a typical hydrothermal method, and the synthesis procedures can be proximately described as follows: 0.114g of $\text{In}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (99.99 purity) and 0.4g of urea were dissolved in 20 mL deionized water, and stirred at room temperature for 30 min. The resulting mixture was transferred and sealed in a Teflon-lined stainless steel autoclave (25 mL capacity). After placing the autoclave at 180°C for 5 h followed by cooling the autoclave down to room temperature naturally, a white precipitate was collected and washed with deionized water and then absolute alcohol. The washing cycle was repeated three times, followed by drying at 60°C for 1 h. The products were white $\text{In}(\text{OH})_3$. Finally, the

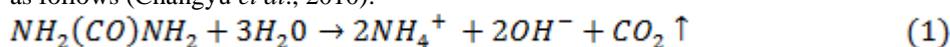
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obtained products were calcined in air at three temperatures 300,400 and 500 °C for 2 h with a heating rate 5 °C·min⁻¹ and cooled under air. The products were In₂O₃ nanostructures. For the synthesis of films In₂O₃ the as preparation powder was dissolved in (2ml) ethanol with a constant stirring then dropped on a glass slide and heated to temperature 50 °C for 30 min . Finally, the obtained films were calcined at 500 °C for 2 h with a heating rate 5 °C /min.

The particle size and morphology were examined by using field emission scanning electron microscopy (FE-SEM) (Hitachi-S4160).. The crystalline phase was examined by X-Ray diffraction (XRD) cu α ($\lambda= 1.54 \text{ \AA}$) radiation with 2θ ranging (20-70).The optical properties of In₂O₃ nanostructure was investigated using (double beam spectrophotometer UV-210A Shimadzu). The transmittance and absorbance of In₂O₃ samples prepared at different temperatures have been recorded in the wavelength rang (300-800) nm.

RESULTS AND DISCUSSIONS

Hydrothermal reaction of an aqueous solution of In(NO₃)₃ and urea NH₂(CO)NH₂ leads to the formation of In(OH)₃ nanostructures. Urea plays a significant role in the formation of In(OH)₃ nanostructures. It is well known that temperature-dependent urea hydrolysis is a mild process to produce (OH)⁻, with which the metal ions can precipitate at a slow growth rate via homogeneous nucleation. The reaction process could be described as follows (Changyu *et al.*, 2010):



Heat treatment of In(OH)₃ will result In₂O₃ .



Fig.1. displays XRD patterns of In₂O₃ synthesized at different calcination temperatures. The intensities and locations of the peaks were identified as a cubic structure of In₂O₃ (JCPDS card 6-0416). No impure peaks were observed in the XRD patterns, which indicated high purity of the sample. From the patterns, it is perceptible that as the calcination temperature increased, the intensities of peak (222) to (622) were enhanced. The mean crystallite size D was calculated using Scherer's formula [$D = K\lambda / (\beta \cos \theta)$] Where K is constant equal to (0.9), θ is scattering angle value λ : wavelength of the X-rays (1.5406 Å) and β is full width at half maximum (FWHM) (Raad and Osama, 2014). The crystallite size was found to be (16.11)nm and these value increases to (18.23)nm with an increasing in the calcination temperature from 300°C to 500°C which is due to the sufficient increase in the supply of thermal energy for crystallization. The lattice constant was calculated it was found to be (10.1)nm.

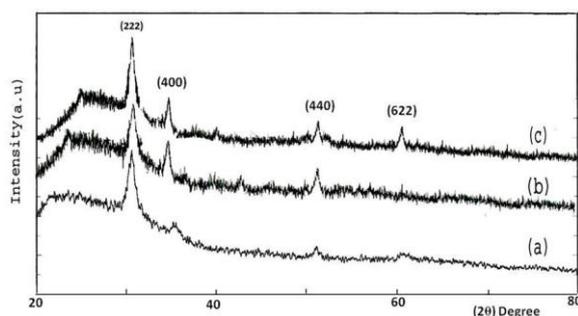


Fig. 1: XRD pattern of In₂O₃ prepared at temperatures (a) 300°C, (b) 400°C, (c) 500°C.

Fig.2. presents the FE-SEM images of In₂O₃ nanostructures prepared at different calcination temperatures. The results indicate that In₂O₃ nano particles with diameter (64.45)nm were grown at calcination temperature 300°C Fig.2a .When the temperature increases to 400°C , In₂O₃ nanorods were observed clearly with diameter being about (90)nm Fig.2b .Also a micro cubic were observed When the calcination temperature rose to 500°C Fig.2c .Therefore it could be concluded that the calcination temperature was a key factor influencing the size and shape of In₂O₃ nanostructures.

The transmission spectra of In₂O₃ nanostructures prepared at different calcination temperature and measured in the wavelengths between 300 and 800 nm are shown in Fig.3. the spectra displayed a transmittance higher than 80% in the visible region .The optical transmission decreases sharply near the absorption band edge

of In_2O_3 . The optical absorption edge shifted toward high energy as the temperature decreased this behavior is due to the quantum confinement effect. The optical band gap energy as a function of temperature is shown in Fig. 4. the optical band gap was found to be (3.79)nm for sample prepared at calcination temperature 300°C and these value decreases to (3.72)nm with the increasing of calcination temperature to 500°C . This suggests that the density of defects decreases with an increase in the calcination temperature also this behavior is due to the increases of crystal size with the increasing of calcination temperature(Zhenmin *et al.*, 2013).

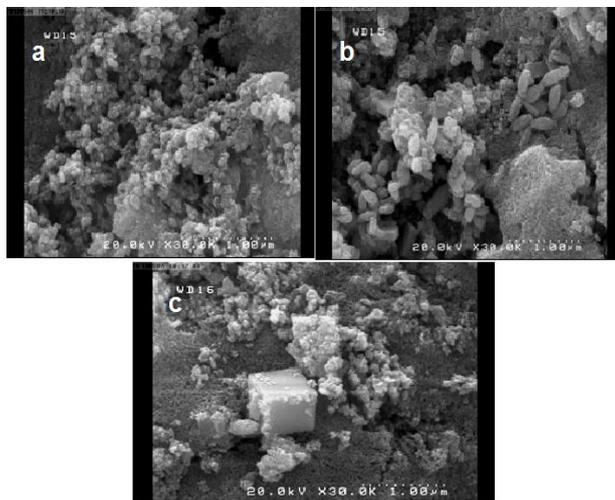


Fig. 2: FE-SEM images of In_2O_3 prepared at temperatures (a) 300°C , (b) 400°C , (c) 500°C .

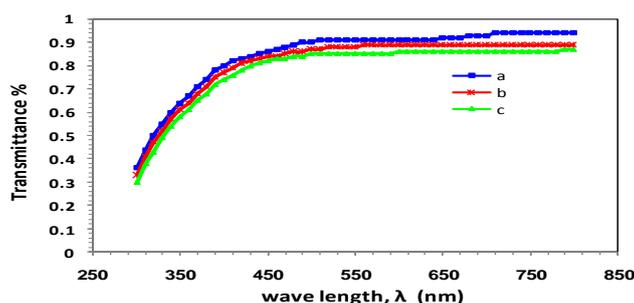


Fig. 3: The optical transmittance of In_2O_3 prepared at temperatures (a) 300°C , (b) 400°C , (c) 500°C .

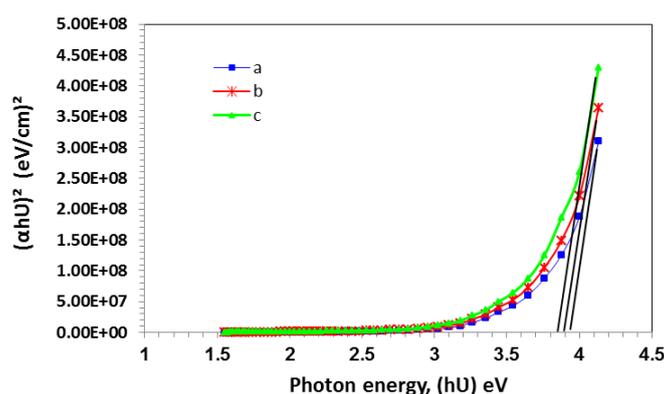


Fig. 4: The optical band gap of In_2O_3 prepared at temperatures (a) 300°C , (b) 400°C , (c) 500°C .

Conclusions:

In summary, In_2O_3 nanostructures both in nanoparticles, nanorods and nanocubic were synthesized by simple hydrothermal method. The effect of calcination temperature were studied, the characterization results show that the crystal size increases with the increasing of temperature. The SEM images shows that a nanoparticles were grown at 300°C , with the increasing of calcination temperature to 400°C a nanorods were

grown, also a nanocubic were grown at 500 C°. The optical band gap was found to decrease with the increasing of growth temperature.

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