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Synthesis of nano-sized indium oxide (In_2O_3) powder by a polymer solution route

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Abstract

Indium oxide (In_2O_3) is a n-type semiconductor with various applications in thin film coatings, on the basis of its optical properties, and in gas sensing equipment, due to its high sensitivity to various oxides such as CO_x and NO_x. In this study, a synthesis process for obtaining In_2O_3 nanoparticles is examined. The precursor used is indium nitrate hydrate ($InN_3O_9 \cdot H_2O$) because of its high solubility in water. By dissolving the nitrate salt in a PVA (polyvinyl alcohol) solution, the precursor is dispersed homogeneously, which reduces the agglomeration of the resulting powder. Calcination at a low temperature of 200–250 °C burns out the organic materials of the PVA with NO_x gas emission and allows the oxidation of the indium, resulting in indium oxide nanoparticles. The influence of the PVA solution characteristics and the heat treatment temperature on the powder morphology and size was analyzed by using SEM, TEM, XRD, TGA/DSC, and four point BET for a specific surface area analysis. The measured specific surface area varies from 3 m^2/g to 76 m^2/g depending on the calcination temperature, and the particle size of the synthesized powders is under 10 nm for the samples heat treated at 300 °C.

Keywords: A. Calcination; A. Grain growth; B. Porosity; In_2O_3

1. Introduction

Indium oxide (In_2O_3) is a n-type semiconductor with a band-gap of 3.55–3.75 eV, which is very close to that of GaN (~3.4 eV) [1]. The material has scientific significance due to its interesting properties such as high transparency to visible light and high electrical conductance and because of its strong reaction when exposed to various gas molecules such as CO_x and NO_x, thus making it very useful for gas sensing equipment [2–4]. Besides its gas sensing applications, In_2O_3 is also used in various fields such as solar cells [5], organic light emitting diodes [6], and photo-catalysts [7], as well as in field emissions [8].

In_2O_3 nanoparticles have been synthesized through various methods including pulse laser deposition [9], thermal hydrolysis [10], spray pyrolysis [11], a sol–gel technique [12], and non-aqueous synthesis [13], and in various morphological nanostructures such as nanocrystals, nanowires, nanorods, and nanobelts [14–17]. The most common synthesis process used is the hydrothermal technique which relies on elevated pressures and temperatures.

In this study, the possibility of synthesizing In_2O_3 nanoparticles by using a polymer solution route is examined. The purpose of using a polymer solution is to ensure minimum agglomeration of the powder during synthesis and also to maintain a high purity of the powder through the entire synthesis process. The high purity is maintained by the complete burn-out of the polymer through calcination. In addition, because the polymer coats the nucleated indium particles, it also inhibits grain growth which helps to obtain a very fine nanosized powder.

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Table 1
Powder synthesis parameters.

Sample	PVA type (MW)	Calcination temperature (°C)
(a)	9000–10000	300
(b)	9000–10000	500
(c)	9000–10000	800
(d)	146000–186000	300
(e)	146000–186000	400
(f)	146000–186000	500
(g)	146000–186000	600
(h)	146000–186000	700
(i)	146000–186000	800

For this study the polymer employed is polyvinyl alcohol (PVA) to make the solutions for dissolving the powder. The influence of changing the PVA molecular weight on the particle morphology and characteristics is also analyzed. For this purpose low and high molecular weight PVA solutions were used.

The benefits of using the polymer solution route for synthesis of ceramic powders are shorter synthesis time and low calcination temperature which result in less energy consumption for material synthesis, and the resulting material shows improved characteristics and high purity [18]. One of the main purposes for using PVA as the organic carrier is that it has a relatively low decomposition temperature and the organic compound can be completely burned out between 200 °C and 300 °C. The polymer solution route has been used to obtain various nano-sized materials with high purity such as yttrium phosphate (YPO_4) and yttria (Y_2O_3) [19,20], and various calcium based complex materials such as tricalcium silicate (Ca_3SiO_5) and tricalcium aluminate ($Ca_3Al_2O_6$) [21], by using long chain polymers such as polyvinyl alcohol or polyethylene glycol.

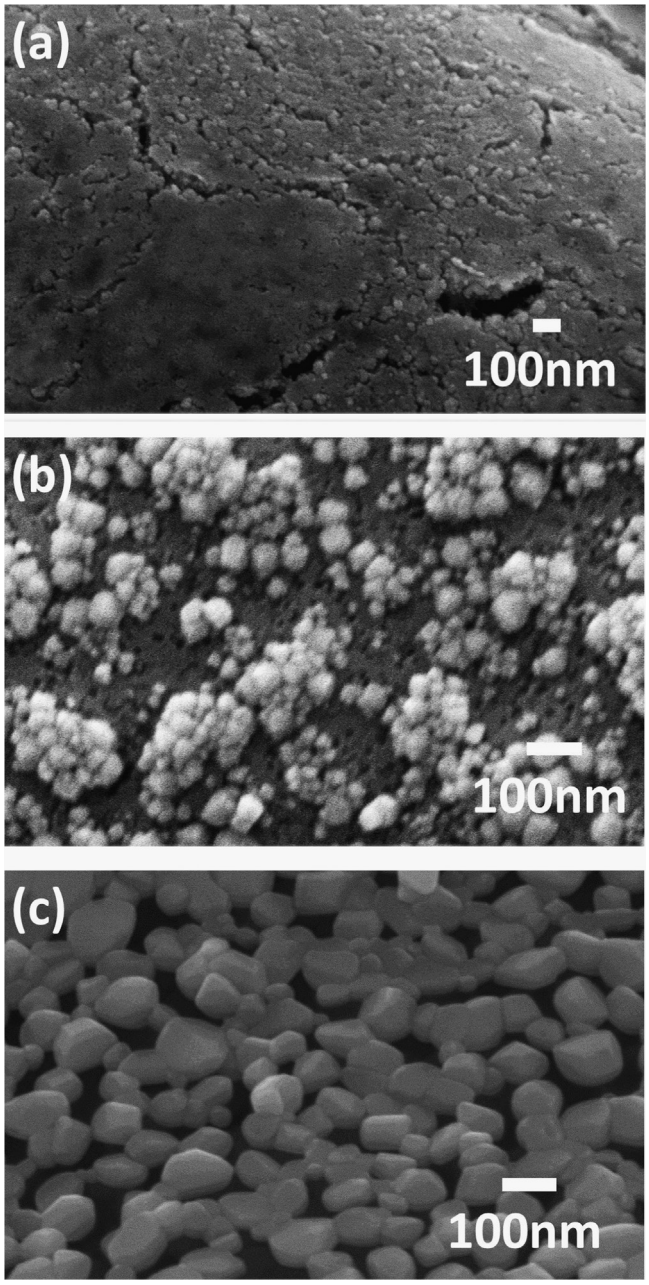

Fig. 1. SEM micrographs of indium oxide powders synthesized by using 5 wt% Low MW PVA solution: (a) 300 °C, (b) 500 °C, (c) 800 °C.

2. Experimental procedure

The synthesis process employed for obtaining In_2O_3 nanoparticles involves the use of deionized water, polyvinyl alcohol (PVA, MW 9000–10000, 80% hydrolyzed and MW 146000–186000, 99% hydrolyzed, Aldrich Chemicals), and indium nitrate hydrate ($InN_3O_9 \cdot H_2O$, Aldrich Chemicals).

The indium nitrate is dissolved in deionized water at 120 °C with continuous stirring. After the salt is completely dissolved, a 5 wt% PVA solution is added and the temperature is raised to 210 °C and mixed until the water is evaporated. The mixing speed was maintained at 150 rpm throughout the heating on the hot plate. The resulting compound is put in a drying oven for 24 h at 100 °C in order to remove any residual water left inside. After drying the powder is calcined in order to eliminate the organic carriers and to oxidize the indium powder. The calcination was conducted at a heating rate of 3 °C/min and a holding time of 1 h.

Two types of PVA were used to obtain the polymer solution. The amount of PVA to indium salt was maintained at a ratio of 8:1 of positive charged ions from the indium precursor to the negative charged ions from the PVA monomers. The PVA to indium salt ratio was also varied as 2:1 and 4:1 but in these cases the carbon was not fully removed until the heat treatment temperature exceeded 500 °C.

The synthesis parameters of each of the powders can be observed in Table 1. During the mixing on the hot plate, after