

# Synthesis and characterization of indium nanoparticles

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**Abstract.** In this paper, we present the results on synthesis of indium nanoparticles by the reduction of indium chloride in a solution of sodium borohydride ( $\text{NaBH}_4$ ). The indium nanoparticles were characterized by X-ray diffraction, transmission electron microscopy (TEM) and UV-visible spectroscopy. The analysis of X-ray diffraction (XRD) indicates that the indium nanoparticles possess a tetragonal crystal structure. The TEM images demonstrate that the nanoparticles with a size about 100 nm were agglomerate from many smaller particles. Effect of some technology conditions on the formation of the indium nanoparticles was studied.

## 1. Introduction

Nanostructured materials are an important research issue of science and technology. Those materials have some new characteristics compared with the bulk materials; these properties can bring various promising applications in science and technology and in the life as well. By changing technological conditions, one can control the size of the particles and hence obtain new unique properties due to the quantum confinement effect.\*

Metal nanoparticles have attracted a great deal of attention because of their diverse application in electronics and bionanotechnology [1,2]. Gold [3], silver [4] are among the most useful metals suited for the nanoparticles synthesis for electronics and bionanotechnology. The nanoparticles based on other metals like Co [5], Sn [6], Fe [7], Ni [8] and In [9] have been also studied. Recently, it was found that the indium (In) nanoparticles have been explored for their potential applications in electronics (for single electron transistor), bionanotechnology (as tags for detection of DNA hybridization) [10,11] and as starting material for convenient synthesis of InP using phosphide ions

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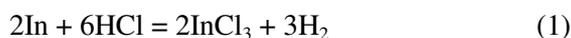
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[12]. The In nanoparticles have previously been synthesized by vapor phase deposition method [13], electrochemical reduction [14], chemical reduction of salts [9] laser ablation [15] and ultrasound irradiation [16].

In this paper, we describe the results on the preparation of indium nanoparticles by a chemical reduction of indium chloride salts in a solution of sodium borohydride ( $\text{NaBH}_4$ ). The In nanoparticles were characterized by XRD, TEM, energy-dispersive X-ray (EDX) spectroscopy and UV-vis absorption spectra.

## 2. Experimental

For preparation of indium chloride, 1g of indium metal was mixed with 2 ml chlorhydric acid HCl (36.5%). Then, the solution was heated at  $70^\circ\text{C}$  for 2 h. Reaction equation is as follows



After reaction finished, 100 ml of pure water was added into the above solution. Final solution  $\text{InCl}_3$  was called original solution. Adding pure water to 1.437 ml of original solution, one obtained 5 ml of  $\text{InCl}_3$  25 mM solution (called as solution A). 0.25 g of poly(vinyl pyrrolidone) (PVP) was dissolved in 5 ml of pure water to product solution B. Dissolving 28.5 mg  $\text{NaBH}_4$  in 5 ml of pure water, one produced  $\text{NaBH}_4$  0.15 M solution (called as solution C).

Solution A and solution B were mixed and stirred at room temperature for 10 min., and then solution C was poured into the mixture solution in constant stirring. The reaction was carried out at temperatures of  $30^\circ\text{C}$ ,  $50^\circ\text{C}$  and  $80^\circ\text{C}$ , and was described by the following equation:



After 10 min of stirring at  $50^\circ\text{C}$  and  $80^\circ\text{C}$ , the solution became a black liquid. This liquid was centrifuged with 9000 rpm to obtain a black powder material which was dried and crushed. Scheme of synthesis procedure of indium nanoparticles is shown in Fig. 1.

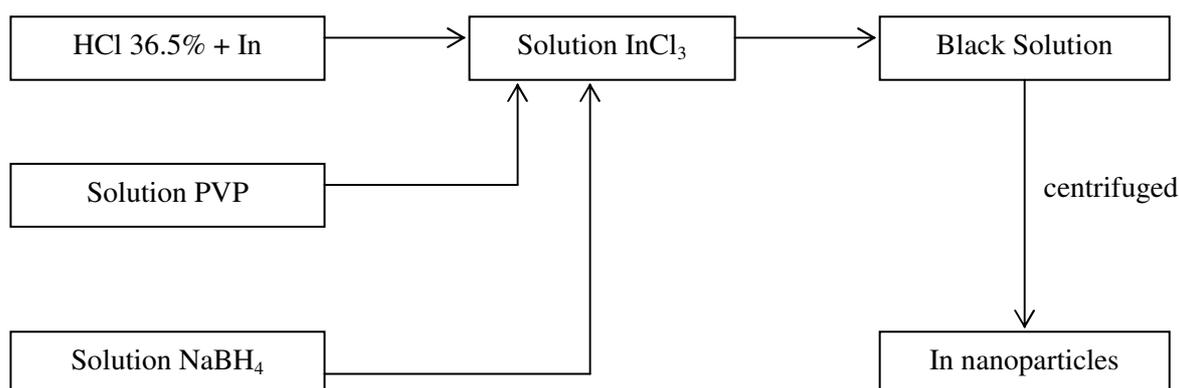


Fig. 1. Scheme of synthesis procedure of In nanoparticles.

The In nanoparticles crystalline structure was examined by XRD using a Bruker D5005 diffractometer. The morphology of the In nanoparticles was observed by TEM using a JEOL JEM

1010 electron microscope. The composition of the samples was determined by an EDX spectrometer OXFORD ISIS 300 attached to the JEOL-JSM 5410 LV scanning electron microscope. The absorption spectra of the In nanoparticles were recorded by a Shimadzu UV 2450 PC spectrophotometer.

### 3. Results and discussion

In this report, our research focused on the influence of reaction temperature and concentration of  $\text{NaBH}_4$  on the nanoparticle crystalline structure. In these experiments, the content of  $\text{InCl}_3$  and PVP was kept constant, but the content of  $\text{NaBH}_4$  and the reaction temperature were changed as shown in table 1. In the reaction,  $\text{NaBH}_4$  acted an agent to reduce  $\text{In}^{+3}$  to  $\text{In}^0$ ; PVP played the role of surfactant covering the indium nanoparticles to avoid agglomerate and oxidation of indium particles.

Table 1. The content of precursors and reaction temperatures for different samples.

Sample	$\text{InCl}_3$ solution (ml)	PVP solution (ml)	$\text{NaBH}_4$ solution (ml)	Reaction temperature ( $^{\circ}\text{C}$ )
M1	5	5	5	30
M2	5	5	7,5	30
M3	5	5	10	30
M4	5	5	5	50
M5	5	5	7,5	50
M6	5	5	10	50
M7	5	5	5	80
M8	5	5	7,5	80
M9	5	5	10	80

TEM images of the as-prepared In nanoparticles in samples M7 and M8 are shown in Fig. 2. It can be seen that the In particles having an average size of about 100 nm are agglomerated from small crystallites.

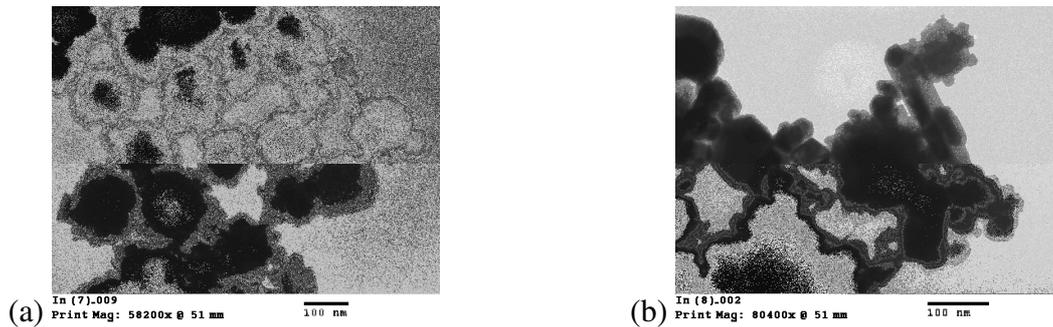


Fig. 2. TEM images of (a) sample M7 and (b) sample M8.

XRD pattern of the indium nanoparticles in sample M7 is shown in Fig. 3. From the figure it is clear that the XRD pattern exhibits prominent peaks at  $2\theta$  angles of  $32.96^\circ$ ,  $36.70^\circ$ ,  $39.17^\circ$ ,  $54.48^\circ$  and  $56.58^\circ$ , which correspond to the (101), (002), (110), (112) and (200) diffraction planes, respectively. All the diffraction peaks can be perfectly indexed to the indium metal with the tetragonal phase structure. Lattice constants were determined from XRD pattern to be  $a = 3.251 \text{ \AA}$  and  $c = 4.940 \text{ \AA}$  in good agreement with the well-known lattice parameters for bulk indium:  $a = 3.251 \text{ \AA}$  and  $c = 4.945 \text{ \AA}$  (JCPDS 85-1409). The information on the crystalline size has been obtained from the following Debye - Scherrer relations [17]:

$$L = 0.9\lambda / (\beta \cos \theta) \quad (3)$$

where  $\beta$  is the full width at half maximum (FWHM) in radians of the diffraction peaks,  $\theta$  is the Bragg's diffraction angle and  $\lambda$  is the wavelength for the  $K_{\alpha 1}$  component of the employed copper radiation ( $1.54056 \text{ \AA}$ ). The size of the indium nanoparticles in sample M7 is estimated to be 110 nm in agreement with the results of TEM analysis.

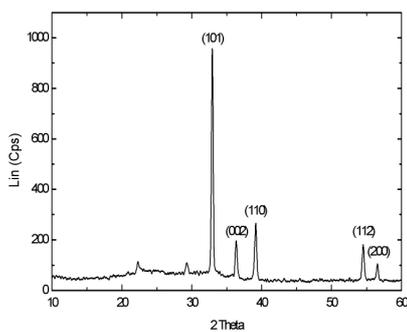


Fig. 3. XRD pattern of In nanoparticles in M7 sample.

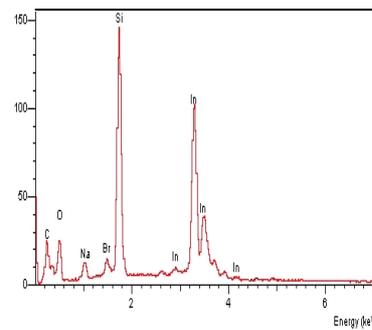


Fig. 4. EDX spectrum of In nanoparticles in M7 sample.

EDX spectrum of sample M7 shown in Fig. 4 indicated that the main composition of the samples was In metal, Si element was from silicon substrate which was covered with the layer of the measured In nanopowder; the elements C, O, Na, Br were from the precursor solution.

UV-vis absorption spectrum of the indium nanoparticles is presented in Fig. 5. The spectrum exhibits a characteristic surface plasmon absorption band centered at 300 nm. The surface plasmon absorption bands centered at 238 nm [18] and 240 nm [19] were observed for the individual indium nanoparticles less than 50 nm in size. Meanwhile, the authors [9,15] reported that the surface plasmon absorption bands observed in their indium nanoparticle samples were located at around 400 nm and 350 nm, respectively.

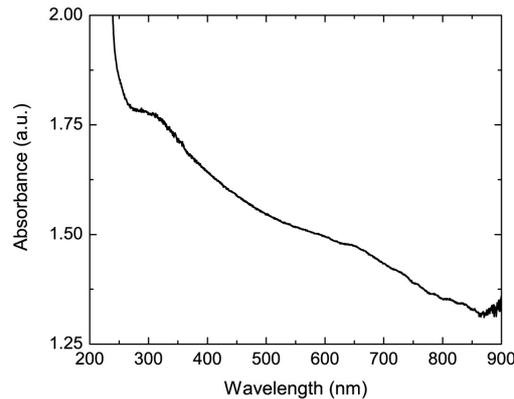


Fig. 5. UV-vis absorption spectrum of the as-prepared indium nanoparticles dispersed in PVP.

Effect of the reaction temperature on the formation of indium nanoparticles was investigated in detail. In these experiments, the content of the precursor chemicals was kept constant, while the reaction temperature was increased from 30°C to 50°C and 80°C. The results are described in Fig. 6. For sample M1 prepared at 30°C (Fig. 6a), the XRD pattern showed a diffraction peak at 2θ-value of 22.53° probably originating from In(OH)<sub>3</sub> [20] and two peaks at 30.58° and 51.10° corresponding to the (222) and (440) crystal planes of In<sub>2</sub>O<sub>3</sub>, respectively (JCPDS 01-0929). Almost no any peak for indium metal appeared. For sample M4 prepared at 50°C (Fig. 6b), in the XRD pattern beside a peak from In(OH)<sub>3</sub> and three peaks at 30.58°, 45.44° and 51.10° which match the (222), (431) and (440) crystal planes of In<sub>2</sub>O<sub>3</sub>, five peaks at 32.96°, 36.70°, 39.17°, 54.48° and 56.58° corresponding the (101), (002), (110), (112) and (200) lattice planes of indium metal appeared.

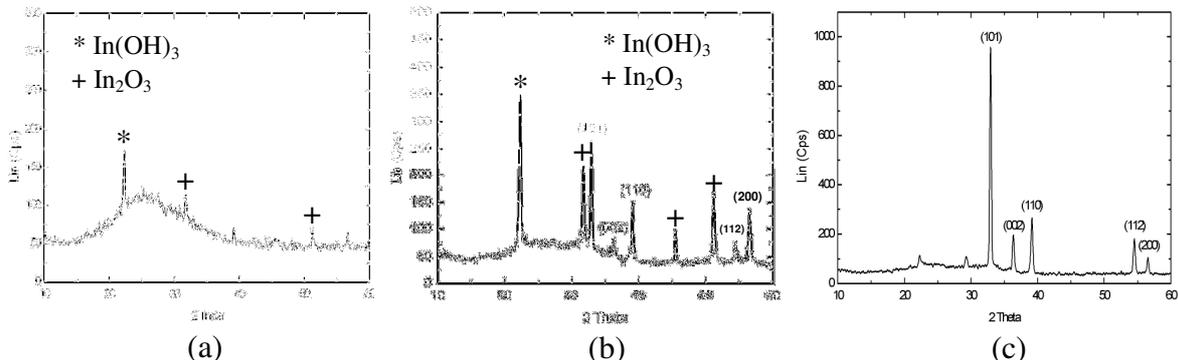


Fig. 6. The XRD patterns of (a) samples M1 prepared at 30°C, (b) samples M4 prepared at 50°C and (c) samples M7 prepared at 80°C.

For sample M7 prepared at 80°C (Fig. 6c), the peaks from indium metal appeared with a very strong intensity (note the scale values). All the diffraction peaks in the XRD pattern were perfectly indexed to the indium metal with the tetragonal phase structure as above presented.

Effect of NaBH<sub>4</sub> concentration on the formation of indium nanoparticles was studied as well. In these experiments, the reaction temperature was kept constant at 50°C, while the content of NaBH<sub>4</sub> solution was increased from 5 ml, 7.5 ml and 10 ml. The results are described in Fig. 7. For sample M4 prepared with 5 ml of NaBH<sub>4</sub> solution, (Fig. 7a), the XRD pattern exhibits a peak from In(OH)<sub>3</sub>, three peaks at 30.58°, 45.44° and 51.10° from In<sub>2</sub>O<sub>3</sub> and five peaks 32.96°, 36.70°, 39.17°, 54.48° and 56.58° from indium metal. When increasing the content of NaBH<sub>4</sub> solution up to 7.5 ml (Fig. 7b), the peak from In(OH)<sub>3</sub> markedly decreased in intensity; two peak at 30.58° and 51.10° from In<sub>2</sub>O<sub>3</sub> disappeared, while the intensity of the peak at 45.44° clearly decreased. When the content of NaBH<sub>4</sub> solution increased up to 10 ml (Fig. 7c), the diffraction peaks corresponding the (101), (002), (110), (112) and (200) lattice planes of indium metal were dominant.

The above-mentioned results allow us to conclude that the reaction temperature and the content of reduce agent NaBH<sub>4</sub> play an important role in formation of indium nanoparticles.

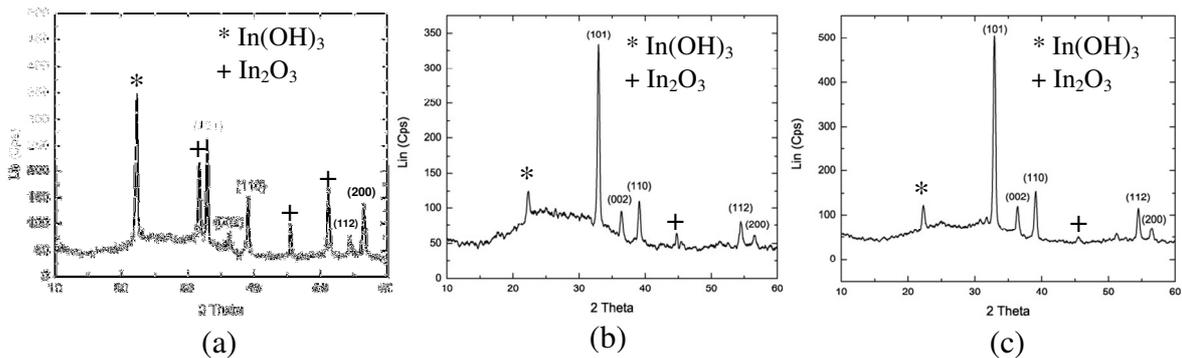


Fig. 7. The XRD patterns of (a) samples M4 prepared with 5 ml of NaBH<sub>4</sub> solution, (b) samples M5 prepared with 7.5 ml of NaBH<sub>4</sub> solution and (c) samples M6 prepared with 10 ml of NaBH<sub>4</sub> solution. The reaction temperature was 50°C.

#### 4. Conclusion

We have successfully synthesized indium nanoparticles by the reduction of indium chloride in a solution of sodium borohydride NaBH<sub>4</sub>. The indium nanoparticles possess the tetragonal crystalline structure. Lattice constants were determined from XRD pattern to be  $a = 3.251 \text{ \AA}$  and  $c = 4.940 \text{ \AA}$ . The average size of indium nanoparticles was determined from TEM and XRD analyses to be about 100 nm. The characteristic surface plasmon resonant absorption band located at 300 nm. It was found that the reaction temperature and the content of reduce agent NaBH<sub>4</sub> play an important role in formation of indium nanoparticles.

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