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Influence of the annealing conditions on the properties of InP thin films

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Abstract

InP thin films were prepared on glass substrates by the spray pyrolysis technique. Results on structural, optical and electrical properties of the layers as a function of the thermal annealing are reported. XRD data indicates that samples show microstructural perfection improvement as a function of annealing time. The optical band-gap shows a dependence with the inverse of the squared crystallite size, suggesting that electron confinement has some effect. The lattice parameter and band-gap energy (BGE) of the samples annealed at 450 °C for 160 min (20–40–80–120–160–200) correspond to the reported values of InP thin films. In addition, the electrical resistivity measurement shows a slight decrease when annealing time is increased up to 80 min but it saturates annealing times between 80 and 160 min. Then it is slightly increased for larger times.
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1. Introduction

The use of wide band-gap III–V compound semiconductors for the fabrication of various electronic and optoelectronic devices has received growing interest in the last years. InP is an important semiconductor material for the fabrication of optoelectronics, microwave devices, and integrated circuits used in modern high-speed optical communication system. The widespread use of InP-based devices in lightwave communication systems has resulted in intensive research on InP and related compound semiconductor materials grown using a number of techniques. Conventionally, InP material is grown by metalorganic vapour phase epitaxy [1,2] (MOVPE), gas source molecular beam epitaxy [3,4] (GSMBE) and chemical beam epitaxy [5] (CBE). These techniques have been successful for the growth of high quality InP epitaxial layers, but published work on the preparation and characterization of InP thin films grown spray pyrolysis is still very limited.

InP, with direct band-gap at 1.34 eV, is a very important material in manufacturing light emitting diodes and high mobility transistors. Also a lot, more and more researches have been reported [6,7]. Kaner and co-workers have reported a conventional solid-state metathesis method to synthesis binary III–V semiconductors by reacting sodium pnictides with metal halides in either bombs or sealed glass ampules at high temperatures [8]. However, this route requires rather harsh conditions and high temperatures. Kher and Wells investigated a similar route by the reaction between $(Na/K)_3P$ and $InCl_3$ to prepare InP nanocrystals [9]. Recently, monodispersed InP nanocrystals were synthesized via the reaction of $InCl_3$ and $P(Si(CH_3)_3)_3$ [5]. Qian and co-workers have prepared InP nanocrystals by solvo-thermal and sonochemical methods, respectively [10,11]. Although a lot of investigations on the preparation of III–V semiconductor crystals have been done, few were concerning the reaction mechanism. Here in this work, we report a new method to prepare InP thin films by the reaction of $InCl_3$ and Na_2HPO_4 and also the variation of the properties of the samples as a function of the annealing time in air ambient.

2. Experimental details

A double nozzle sprayer made of glass was designed and fabricated in our laboratory to prepare thin film samples by spray pyrolysis technique. It is a coaxial assembly of two corning glass tubes, in which the diameters of inner and outer tubes are 6 and 14 mm, respectively. Both the tubes were tapered at one end with a tapering angle of 30° to form the spray nozzle. Well-cleaned and degreased glass substrates were kept inside a tubular furnace designed for this technique. The furnace was resistively

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heated with kanthal wire and the temperature was controlled by a dimmerstat. A temperature controller automatically maintained the required temperature in which the setting could be made manually. A chromel–alomel thermocouple was used to sense the temperature inside the oven, which was in contact with the substrate holder. Inner tube of the spray nozzle was connected to the air compressor and the outer tube to the solution reservoir. The pressure of the carrier gas was monitored through the valve flow meter set. The solution flow rate was determined with the help of a stop clock and a graduated burette as the reservoir. The deposition conditions were optimised in order to obtain reproducible and good quality films. Its colour, adhesion, surface smoothness and chemical treatment determine the quality of the film. Purified compressed air at a pressure of $10^4\,Nm^{-2}$ was used as the carrier gas. The nozzle-to-substrate distance was approximately 25 cm. High purity $InCI_3$ and Na_2HPO_4 were dissolved separately in a solution containing deionised water. The molarities of $InCI_3$ and Na_2HPO_4 solutions were 0.5 and 0.05 M, respectively. The substrate temperature was maintained at 500 °C. The flow rate of the solution during spraying was adjusted to be $0.5\,ml\,min^{-1}$ and kept constant throughout the experiment.

Post-deposition thermal treatments of the samples were performed in an air atmosphere at a fixed temperature of 450 °C for periods in the 20–200 min range. For these thermal treatments we proceeded as follows: the samples were divided into two parts, each with the same size. One of them was annealed during 20 min (called first annealing); then the same sample was made during 40 and 80 min (called second and third annealing). For the second part, a fourth, fifth and sixth annealing were made during 120, 160 and 200 min, respectively. After each annealing the structural, optical and electrical properties were measured. The layer thickness was measured before and after the annealing by using a step profiler (Sloan Dektak II). No change of the sample thickness was observed within the resolution of the equipment. Optical transmission data were obtained with an Perkin Elmar Lambda 25 UV–vis-NIR double-beam spectrophotometer in the 300–900 nm range. The crystalline structure of the films was analyzed with a Rigaku D/Max-IIIS model X-ray diffractometer ($\lambda = 1.5405$ Å) using the Cu Kα line. The diffractograms were smoothed using the DIFRAC-AT program [12]. The maximum was fitted by means of the FULLPROF program [13] using a pseudo-Voigt function [14,15]. From these fittings, the integral width (β) and the full width at half maximum (FWHM) were obtained. Then, with the aim of determining the tendencies of the crystallite size and microstrain, a Willamson Hall graphics [16,17] was built. The crystallite size and the microstrain were determined using the method of the single line [18], averaging the best quality measured lines.

3. Results and discussions

Fig. 1 shows the X-ray diffraction profiles of InP thin films with different annealing times. For the as-deposited films the peaks associated to planes (1 1 1), and (2 0 0) of the cubic structure are observed. When the samples are annealed, the results can be divided into two categories: (a) annealing times for which

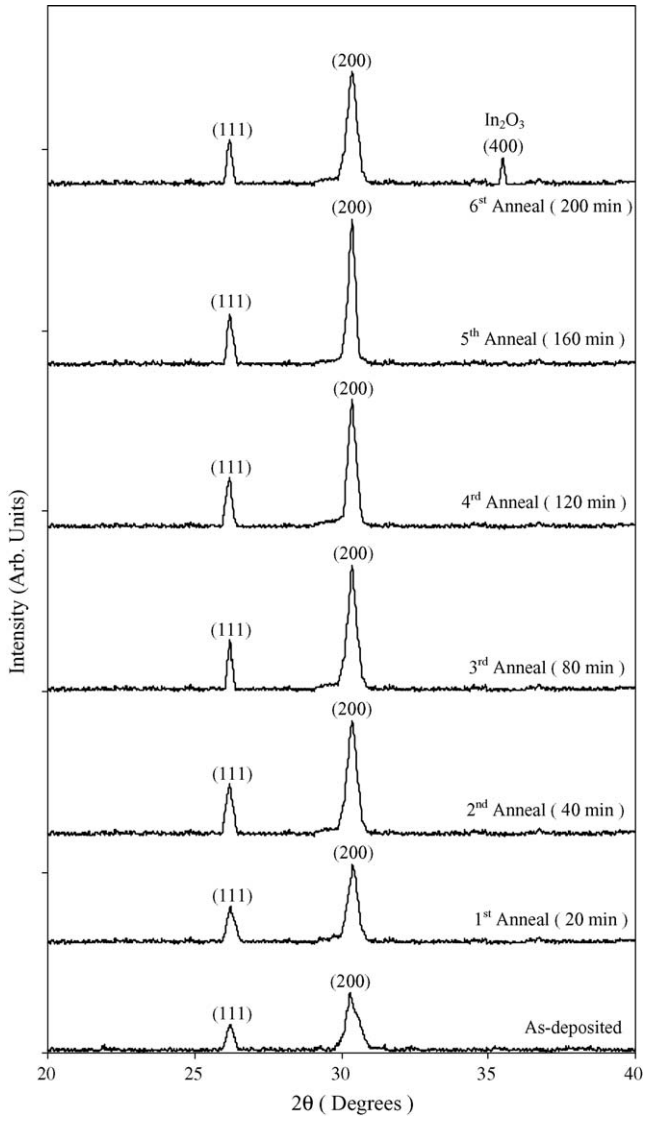

Fig. 1. X-ray diffraction patterns of InP thin films at the substrate temperature 500 °C. The annealing was carried out at 450 °C in air for different times (a) 20 min; (b) 40 min; (c) 80 min; (d) 120 min; (e) 180 min and (f) 200 min.

there is an increase of the intensity of the peaks (crystallites are oriented with the [2 0 0] direction perpendicular to the substrate, with a slight shift of the maximum towards higher 2θ values), and (b) increasing crystallite size, D and decreasing microstrain, $\langle e \rangle$ values when the annealing time is further increased. Table 1 shows the crystallite size, the microstrain associated to the (2 0 0) peak and the lattice parameter. From these results, we can see that the fundamental effect of the annealing is related to an increase in the size of the crystallites and a decrease in the lattice parameter and the microstrain. As the annealing time increases the intensity of InP (2 0 0) peak increases and this peak becomes narrower indicating an improvement of the crystallinity. This means that the crystallite size of the films increases with increasing up to 160 min annealing time (see Table 1). The only main peak (2 0 0) obtained with all films indicating that the increasing of annealing time does slight change the preferred growth orientation. The (0 0 2) direction corresponds to

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