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The influence of argon pressure and RF power on the growth of InP thin films

G Hema Chandra¹, J Pérez de la Cruz¹ and J Ventura²

¹ INESC Porto, Rua do Campo Alegre 687, 4169-007 Porto, Portugal
² IFIMUP, Rua do Campo Alegre 687, 4169-007 Porto, Portugal
E-mail: drghec@rediffmail.com

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Abstract

Indium phosphide thin films were grown onto glass substrates by RF magnetron sputtering. In this paper, we present a study on the role of argon pressure and rf power on magnetron sputtered InP films. These sputtering parameters are shown to affect the deposition rate, structure, morphology, electrical and optical properties of InP films. Single-phase, nearly stoichiometric and polycrystalline films exhibiting zinc blende structure with strong preferred orientation along (1 1 1) were observed at an argon pressure of 0.4 Pa, by keeping the substrate temperature (448 K) and RF power (150 W) constant. Hall measurements indicated n-type conductivity in InP films. The optical absorption studies indicated a direct band gap of 1.35 eV.

1. Introduction

Indium phosphide has proved to be a promising material for the fabrication of high electron mobility transistors [1], optical gratings [2], semiconductor lasers [3] and high-efficiency solar cells [4]. InP-based ternary and quaternary compound semiconductors such as InGaP, InGaAs and InGaAsP provide several interesting opto-electronic properties suitable for multi-junction solar cell applications [5]. Stability against degradation of solar cells due to radiation is extremely important for space applications. InP reported to have much better radiation resistance than GaAs and Si [6].

Single-crystal InP wafers are undesirable for solar cell fabrication and operation because of high cost, high fragility, high mass density and low thermal conductivity. So the development of suitable techniques for fabricating high-efficiency solar cells is essential. Polycrystalline films are necessary for solar cell fabrication in order to minimize the wastage of material. Considerable attention has been devoted by various authors to the preparation of InP thin films by different methods such as flash evaporation [7], pulsed laser deposition [8, 9], sputtering [10–13], molecular beam epitaxy [14–16], electro-deposition [17], spray pyrolysis [18, 19] metal-organic chemical vapour deposition [20–23], liquid phase epitaxy [24] and organic chemical vapour deposition [25, 26] to study various electro-optical properties for the device applications. Every technique has associated with it its own merits and demerits. The decisive requirements for the efficient performance of the devices are compositional uniformity and crystallinity. One of the main problems in the InP thin-film growth is that phosphorus vaporizes at temperatures considerably below the melting point of InP [27, 28].

Among several thin-film deposition methods, RF sputtering is known to be a suitable technique for large-scale applications in device fabrication. Although there are few reports on InP films by the sputtering technique [10–13], most of them have focused on the effects of the substrate temperature on their growth and properties. However, to the best of our knowledge, no data on the role of argon pressure and RF power on the properties of InP thin films are reported in the literature. Hence, detailed investigation on the impact of process parameters on composition, structural, electrical and optical properties is reported systematically in this study. The results obtained were analysed to evaluate the potential of the RF sputtered InP films for solar cell fabrication.

2. Experimental details

InP thin films were deposited onto ultrasonically cleaned glass substrates by the RF magnetron sputtering system from a commercially available monocrystalline InP (1 0 0) target with 99.999% purity of 2” diameter. The sputtering was conducted in Ar atmosphere with a target-to-substrate distance of 5 cm. After the chamber was evacuated to a base pressure less than 10⁻⁴ Pa, pre-sputtering was carried out for 10 min at an argon...
gas pressure of 1.0 Pa in order to clean the target surface. The substrate temperature was monitored and controlled through a PID temperature controller (Eurotherm 2408) using a k-type thermocouple placed nearer to the substrates. The thickness was kept constant at 800 nm. To optimize the deposition conditions to get nearly stoichiometric, polycrystalline and single-phase InP films, two different sets of films were prepared by changing a specific parameter, with the others remaining constant. The values of the deposition parameters are reported in table 1.

The elemental composition of the films was determined using energy dispersive x-ray (EDX) analysis attached to the JEOL scanning electron microscope (SEM). The optical transmittance and reflectance of the films were recorded at room temperature in the wavelength range 500–1500 nm using an UV–vis–NIR double beam spectrophotometer. X-ray diffraction patterns of the films were recorded with a Philips x-ray diffractometer using CuK\(_\alpha\) radiation (\(\lambda = 0.154\) 06 nm). The pattern was recorded with a scan speed of 0.02 °s\(^{-1}\) in the 2θ range 20–60°. The electrical conductivity and Hall mobility were measured using standard techniques.

3. Results and discussion

3.1. Compositional analysis

The EDX spectra of InP thin films revealed that the In and P contents depend critically on the argon pressure during sputtering. Figure 1(a) shows the variation of elemental atomic percentages of In, P with argon pressure for the InP films of 800 nm thickness. It is seen that InP thin films formed at a pressures above 0.4 Pa contained excess indium, and were deficient in phosphorus. The films grown at a pressure of 0.4 Pa, by keeping the RF power (150 W) and substrate temperature (448 K) constant, are found to be nearly stoichiometric and the calculated atomic percentages (at.%) of indium and phosphorus were 51.91 and 48.09, respectively, which were comparable to the starting material composition (at. %: In = 50 and P = 50) within the limits of the experimental error ± 0.5 at.%. Figure 1(b) shows the EDX analysis spectra of InP films (800 nm) deposited at 0.4 Pa. The P/In atomic ratio is found to decrease from 0.93 to 0.59 with the increase in argon pressure from 0.40 to 1.0 Pa. The variation in the composition with argon pressure is attributed to the scattering effect of unequal atomic masses of In (114.818 amu) and P (30.973 amu) with Ar atoms. During the sputtering process, In and P atoms are scattered by argon atoms. However, this scattering effect is relatively weak for indium atoms due to its large atomic mass. Thus, more indium atoms can deposit onto the substrate with increasing gas pressure, leading to the In-rich InP films.

Figure 1(c) indicates the variation of In and P elemental atomic percentages with RF power by keeping the argon pressure (0.4 Pa) and substrate temperature (448 K) constant. It is observed that the P/In atomic ratio increased from 0.85 to 0.93 with the increase in RF power from 50 to 150 W.
The self-bias voltage is found to increase (285–575 V) with the increase in RF power (50–150 W). The self-bias voltage directly affects the energy of argon ions striking the target and the particles (sputtered atoms and secondary electrons) bombarding the substrate surface. So an increase in RF power not only increases the deposition rate, but also increases the incident energy of the atoms deposited at the substrate [29–31]. The scattering probability of phosphorus atoms with argon atoms is less when they are moving with higher velocities from the target to the substrate (maintaining the pressure and substrate temperature constant). The high deposition rate favours the incorporation of phosphorus into InP films for becoming nearly stoichiometric at the RF power of 150 W.

### 3.2. Structural analysis

Figure 2(a) shows the XRD pattern of InP films deposited by varying the argon pressure (0.4–1.0 Pa), with a constant RF power of 150 W and a substrate temperature of 448 K. The XRD pattern clearly showed the presence of the indium phase along with the InP phase in the layers grown with different sputtering pressures. The films grown at sputtering pressures from 0.6 to 1.0 Pa exhibited two phases, namely InP and In. The peaks located at $2\theta = 26.31$, 30.46, 43.61 and 51.64$^\circ$ were due to the InP phase corresponding to the $(111)$, $(200)$, $(220)$ and $(311)$ orientations, respectively, which are in good agreement with the standard JCPDS data (card no 03-065-5527) and the peaks observed at $2\theta = 33.02$, 36.43, 39.21 and 54.51$^\circ$ were due to the metallic indium phase corresponding to $(101)$, $(002)$, $(110)$ and $(112)$, respectively, which matches with the JCPDS data (card no 01-071-4627). The In phase was found to be suppressed with the decrease in sputtering pressure, and no In phase was observed at 0.4 Pa. Decreasing the pressure will reduce the scattering effect of the sputtered atoms in the gas phase and increase their kinetic energy. The films grown at a pressure of 0.4 Pa ($T_s = 448$ K, $P = 150$ W) were found to be single phased, nearly stoichiometric and polycrystalline, and exhibited a face-centred cubic structure with the strong $(111)$ preferred orientation. The lattice parameter evaluated for nearly stoichiometric InP thin films deposited at 0.4 Pa was found to be $a = 0.5882$ nm. The variation of the lattice parameter and FWHM values of InP $(111)$ with argon pressure is shown in table 2.

The origin of strain is related to lattice ‘misfit’, which in turn depends upon the growing conditions of the films. This can lead to cracking and/or pealing of the film from the substrate. These detrimental effects can directly influence the microstructural properties and thus the optoelectronic
The variation of the strain (%) of films deposited at different argon pressures (0.4–1.0 Pa), maintaining a constant substrate temperature (448 K) and RF power (150 W). The strain (%) was found to show negative values for the films deposited at $P_{Ar} > 0.4$ Pa, indicating compressive strain.

The presence of the indium phase at $2\theta = 32.90^\circ$ associated with (10 1) was clearly observed from the XRD pattern shown in figure 2(c), for the InP films deposited at RF power less than 150 W. The decrease in the peak height of the InP phase along the (1 1 1) direction and increase in the FWHM value (0.38 to 0.43 $^\circ$) with the decrease in RF power from 150 to 50 W are an indication for the decrease in the grain size. Moreover, the position of the XRD peaks shifting to lower 2$\theta$ values with the decrease in RF power is due to incorporation of excess indium into the crystal lattice resulting in an increase in lattice spacing. The variation of the lattice parameter and FWHM values of InP (1 1 1) with RF power is shown in table 3. Figure 2(d) shows the variation of strain (%) with RF power (50–150 W), keeping a constant pressure (0.4 Pa) and substrate temperature (448 K). Strain was observed to be tensile in nature and it decreases with the increase in RF power from 50 to 150 W, due to enhancement in grain growth (as observed from XRD studies).

The lattice spacing is found to increase with the decrease in RF power, whereas it is observed to decrease with the increase in argon pressure with the incorporation of excess indium into grown InP films. For the first case, the variation can be understood on the basis of the ionic radius ($\text{In}^{3+}$ ion: 0.91 Å), since the larger indium ions enter the lattice resulting in expansion of the unit cell by preserving the overall cubic symmetry. However, in the latter case, because of this expansion, either the cubic symmetry may get distorted or the incorporated excess indium dissociates from the lattice resulting in the formation of the secondary elemental phase, in which case the lattice constant is expected to decrease.

### 3.3. Surface morphology

Figures 3(a)–(c) show the surface morphology of InP films deposited at various sputtering pressures, keeping a constant RF power of 150 W and a substrate temperature of 448 K. A correlation between film morphology with composition was clearly observed. The InP films grown with lower sputtering pressure (0.4 Pa) were found to have an improvement in both crystallinity and surface morphology. This can be attributed to the increased kinetic energy and surface mobility of the ad-atoms under low pressures. From figure 3(a), it is observed that the surface morphology of InP films grown at a pressure of 0.4 Pa showing a $P$/In ratio of 0.93 is almost uniform with conical-shaped grains projected vertically, along with few spherical-shaped grains. A similar type of surface morphology was observed for InP nanowires grown by low-pressure metal organic chemical vapour deposition [32]. With the increase in sputtering pressure to 0.6 Pa (shown in figure 3(b)), the surface becomes less dense showing a completely different morphology with coral-shaped grains having tentacles with a spherical tip. In fact, many factors can influence the surface morphology of thin-film materials, such as the nucleation mode, the overlap growth of neighbouring grains, the angular distribution of incident flux and the surface diffusion of ad-atoms, as well as the difference in growth rates between several crystal planes at the surface of the film. The surface appears to be more irregular with the increase in pressure to 1.0 Pa (figure 3(c)). So it is not desirable to operate under conditions (>0.4 Pa) where such features are produced.

Figures 3(d) and (e) are the SEM images showing the surface morphologies of the InP film prepared at the RF power of 100 and 50 W, respectively. The morphology does not vary with change in RF power. But the mean lateral size at the bottom of cones is found to decrease (120–90 nm) with the decrease in the RF power (150–50 W). This is due to the low kinetic energy of ad-atoms striking the substrate and their low surface mobility. Increasing the RF power to 150 W resulted in better surface mobility and a larger grain size, as shown in figure 3(a).

### 3.4. Electrical properties

The electrical resistivity of the InP films formed at different argon pressures (0.4–1.0 Pa), with a constant RF power of 150 W and a substrate temperature of 448 K, is shown in figure 4(a). It varies from 10 to 1.9 $\times$ $10^2$ $\Omega$ cm with increasing argon pressure. It can be seen that the resistivity decreases rapidly by varying the pressure from 0.4 to 0.6 Pa. The decrease in electrical resistivity with the increase in pressure is due to indium segregation to the ridges of the surface leading to the formation of In-rich films (as

### Table 2. The lattice parameter and FWHM of InP films as a function of argon pressure.

<table>
<thead>
<tr>
<th>$P_{Ar}$ ($T_s = 448$ K, $P = 150$ W)</th>
<th>Lattice constant (nm)</th>
<th>FWHM of InP (1 1 1) (deg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4 Pa</td>
<td>0.5882</td>
<td>0.38</td>
</tr>
<tr>
<td>0.6 Pa</td>
<td>0.5863</td>
<td>0.43</td>
</tr>
<tr>
<td>0.8 Pa</td>
<td>0.5862</td>
<td>0.49</td>
</tr>
<tr>
<td>1.0 Pa</td>
<td>0.5859</td>
<td>0.52</td>
</tr>
</tbody>
</table>

### Table 3. The lattice parameter and FWHM of InP films as a function of RF power.

<table>
<thead>
<tr>
<th>$P$ ($T_s = 448$ K, $P_{Ar} = 0.4$ Pa)</th>
<th>Lattice constant (nm)</th>
<th>FWHM of InP (1 1 1) (deg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50 W</td>
<td>0.5952</td>
<td>0.43</td>
</tr>
<tr>
<td>75 W</td>
<td>0.5926</td>
<td>0.42</td>
</tr>
<tr>
<td>100 W</td>
<td>0.5922</td>
<td>0.40</td>
</tr>
<tr>
<td>125 W</td>
<td>0.5916</td>
<td>0.39</td>
</tr>
<tr>
<td>150 W</td>
<td>0.5882</td>
<td>0.38</td>
</tr>
</tbody>
</table>

The behaviour of the layers due to the distorted lattice. The strain in the as-grown films as a function of argon pressure was evaluated using the relation

$$\text{strain(\%)} = \frac{(a - a_o)}{a_o} \times 100\%,$$

where $a$ is the lattice constant of InP films and $a_o$ is the unstrained bulk InP lattice parameter (0.5868 nm), to estimate whether the strain is tensile or compressive in the direction normal to the plane of the films. Figure 2(b) shows the variation of the strain (%) of films deposited at different argon pressures (0.4–1.0 Pa), maintaining a constant substrate temperature (448 K) and RF power (150 W). The strain (%) was found to show negative values for the films deposited at $P_{Ar} > 0.4$ Pa, indicating compressive strain.

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observed in XRD and SEM analysis). Figure 4(b) shows the variation of Hall mobility ($\mu_H$) and net carrier density with the change of argon pressure. It can be seen that the carrier density increased drastically with the increase in argon pressure. The Hall mobility is found to decrease (36 to 9 cm$^2$ (V s)$^{-1}$) with the increase in argon pressure. The decrease in Hall mobility above 0.4 Pa was due to the effects of impurity scattering with the increase in carrier concentration. The electrical resistivity, carrier concentration and mobility values of the InP films deposited at 0.4 Pa ($T_S = 448$ K, $P = 150$ W) are comparable to the reported data of Saitoh et al [33, 34].

**Figure 3.** SEM images of InP films formed at various pressures: (a) $P_{Ar} = 0.4$ Pa, (b) $P_{Ar} = 0.6$ Pa, (c) $P_{Ar} = 1.0$ Pa. SEM images of InP films formed at various RF powers: (d) $P = 100$ W, (e) $P = 50$ W.
The electrical resistivity of the InP films decreased from 10 to 2 Ω cm with the decrease in the RF power from 150 to 50 W by maintaining the argon pressure (0.4 Pa) and substrate temperature (448 K) constant (shown in figure 4(c)). Figure 4(d) shows the variation of Hall mobility ($\mu_H$) and carrier concentration with the change of RF power (150–50 W). The carrier concentration ($1.74 \times 10^{16}$–$5.58 \times 10^{16}$ cm$^{-3}$) and Hall mobility ($36$–$61.5$ cm$^2$ (V s)$^{-1}$) are found to increase with the decrease in RF power. The decrease in electrical resistivity and the increase in Hall mobility with the decrease in RF power (150–50 W) are due to the incorporation of excess indium into the crystal lattice (as observed in EDX and XRD analysis).

The extent of indium incorporation into the crystal lattice plays a crucial role in varying the mobility of InP films deposited by changing process parameters. The P/In atomic ratio decreases from 0.93 to 0.85 with the decrease in RF power (150–50 W) and varies from 0.93 to 0.59 with the increase in argon pressure (0.4–1.0 Pa). Indium acts as a donor in the crystal lattice contributing more free electrons for conduction, resulting in enhancing the mobility and carrier concentration with the decrease in RF power (150–50 W). In the second case, with an increase in the argon pressure above 0.4 Pa, excess indium incorporation in the crystal lattice as an electrically neutral impurity acts as a scattering centre, thus reducing the mobility. All the InP films deposited by changing various process parameters in the present investigation show n-type conductivity, irrespective of composition or crystallization. This might be due to the fact that the phosphorus vacancies act as shallow donors in InP films (P/In ratio < 1).

### 3.5. Optical properties

The transmittance and reflectance spectra for InP films deposited at various argon pressures (0.4–0.8 Pa), with a constant RF power (150 W) and substrate temperature (448 K) are shown in figure 5(a). The films deposited at argon pressures $\geq 0.6$ Pa are neither transparent nor reflective, which might be due to the existence of lattice defects because of excess indium segregation onto the lattice, providing multi-level trap states in the band gap. The optical absorption coefficient ($\alpha$) is obtained from the measured spectral transmittance and reflectance data using the formula

$$\alpha = \frac{1}{t} \ln \left( \frac{(1 - R)^2}{T} \right).$$


where ‘t’ is the thickness of the film. The nature of the optical transition and the optical band gap for each film is obtained from the following equation [35]:

$$\alpha = A(h\nu - E_g)^n / h\nu,$$

where $A$ is a constant. The exponent ‘$n$’ can take values 1/2, 3/2 or 2 based on whether the optical transition is direct allowed, direct forbidden or indirect allowed, respectively. The values of ‘$\alpha$’ are found to obey equation (3) for $n = 1/2$, indicating that the optical transitions are direct allowed in nature. The optical band gap is determined by extrapolating the linear region of the plot $(\alpha h\nu)^2$ versus $h\nu$ and taking the intercept at $\alpha = 0$. Figure 5(b) shows the plot of $(\alpha h\nu)^2$ versus $h\nu$ for the films deposited at a pressure of 0.4 Pa ($T_S = 448$ K, $P = 150$ W). The optical band gaps are found to be 1.35 and 1.23 eV. The former (1.35 eV) corresponds to fundamental band to band transition in InP, while the latter is due to degeneracy giving rise to sub-band transition. A similar trend was observed for InP films grown using a non-aqueous electrolyte by the electro-deposition technique [17]. The fundamental energy gap of 1.35 eV obtained in this study matches the reported data of InP films [10, 11, 22].

The transmittance and reflectance spectra of InP films deposited with varying RF power (50–150 W) by keeping the argon pressure (0.4 Pa) and substrate temperature (448 K) constant are shown in figure 5(c). The variation of energy gap with changing RF power is shown in figure 5(d). The optical band gap found to increase (1.35–1.38 eV) with the decrease in RF power (150–50 W) is attributed to the decrease in particle size (as observed from XRD and SEM analysis) leading to increase in strain. (The highest occupied valance band and the lowest unoccupied conduction band are shifted to more negative and positive values, respectively, resulting in widening of the band gap.)

4. Conclusions

Sputtering parameters are shown to affect deposition rate, structure, morphology, electrical and optical properties of InP films deposited by RF magnetron sputtering. High deposition rates favour the incorporation of phosphorus, whereas, low deposition rates result in excess elemental indium in the grown thin-film samples. The presence of excess indium is a crucial phenomenon leading to adverse growth conditions. The InP films grown at an operating pressure of 0.4 Pa, by keeping $T_S = 448$ K and $P = 150$ W, are found to be nearly stoichiometric and polycrystalline showing face-centred cubic
structure with crystallites preferentially oriented along the (111) direction. All the thin-film samples are showing n-type conductivity, irrespective of composition. The electrical resistivity, mobility and electron concentration of films deposited at a pressure of 0.4 Pa ($T_s = 448 \, K$, $P = 150 \, W$) were found to be $\sim 10 \, \Omega \, cm$, $36 \, cm^2 \, (V \, s)^{-1}$ and $\sim 2 \times 10^{16} \, cm^{-3}$. These nearly stoichiometric, low-resistive and single-phase InP films with an optical band gap of 1.35 eV could be used as an emitter layer in the fabrication of thin-film multijunction solar cells.

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