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Study of the Effect of InP (100) Nitridation Time by XPS Analysis and Electrical Characterisation

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Abstract: This work presents a study of the effect of thin indium nitride (InN) layer deposition time, on InP substrate. The nitrides of group III metals as InN are very important materials due to their applications in optoelectronics (light-emitting diodes and laser diodes). In this paper the nitridation of InP (100) surfaces has been studied in situ using X ray photoelectron spectroscopy (XPS) and ex situ by electrical I-V method in order to determine the thickness and the quality of the elaborated nitride films and the electrical properties of the InN/InP interface. After ionic cleaning by Ar^+ ions, metallic indium crystallites are created, and the nitridation of the substrates is performed using a plasma glow discharge cell produced indium nitride by reaction with these indium clusters. We used the In_{4d} core levels to monitor the chemical state of the surface and the coverage of the species present. We observed the creation of InN bonds while the In-In metallic bonds decrease. The amount of InN varies for each deposition time. A theoretical model based on stacked layers allows us to calculate the thicknesses δ of InN films produces with different evaporation times of N_2.

Keywords: Nitridation; Electrical characterization; InN/n-InP; XPS.

Introduction

The semiconductors containing the III-nitride element have great potential for the micro and optoelectronics devices with semiconductors (Nakamura, 1998). The Indium nitride is the ideal candidate for the manufacture of ultra-high frequency devices, the blue transmitters and the detectors functioning in spectral field UV (Praveen Kumar, 2009). Several methods were proposed for the nitridation of InP surfaces (Maksimov, 2006). In our case, the nitridation is realized with different times in ultra-high vacuum chamber. The process is performed by exposing the InP substrates to a flow of active nitrogen created by a Glow discharge source with high voltage. We will obtain heterostructures with various thicknesses of InN nano-films on InP substrates. The physical properties of these structures were studied using X ray photoelectron spectroscopy (XPS) which reveals to us the composition of the different elements present on InP surface and evaluate the properties and thickness of obtained InN films (Benamara, 2006). The electrical properties of these structures were presented in this paper allowing us to evaluate different parameters such as the saturation current (I_s), the ideality factor(n), the series resistance (R_s), the diffusion voltage (V_d) and the doping concentration (N_d).

Experimental Procedure

S doped InP (100) substrates with thickness of 400±20 µm have been used. The preparation process of the samples is as follows:

- Chemical cleaning ex situ with successive ultrasonic baths (Khediri, 2021) (H_2SO_4, 3% bromine methanol deionized water) before introduction in an ultrahigh vacuum chamber (10^{-6}_ 10^{-7} Pa).
- In situ cleaning by Ar^+ ion beam during 15 minutes (ion energy, 300 eV; sample current, 2 uA/cm^2; at 6x10^{-5} Torr) in UHV chamber (Talbi, 2013) leads to two phenomena, the removal of the contamination layers mainly due to carbon species (Talbi, 2013), and the creation of metallic indium droplets in well controlled quantity by preferential phosphorus sputtering (Akkal, 2000; Sze, 1981).
- The nitridation process has been performed by plasma discharge cell. In this kind of nitrogen source, continuous plasma is produced by a high voltage (about 2.2KV). The substrate is heated at 250°C during the nitridation process, according to previous work investigating the influence of the temperature on the nitridation process (Pan, 1996). The pressure of nitrogen flow inside the chamber is about 10^1 Pa.

Three series of samples are studied in our case. The preparation conditions of the samples are as follow:

- Sample A1 with doping concentration of N_d= **4.6×10^{15}** cm^{-3}: nitridation during 40 min with nitrogen flow angle of 45°.
- Sample A2 with N_d= **$4\text{-}7\times10^{16}$** cm^{-3}: nitridation with nitrogen evaporation time of 45 min and flow angle of 45°.
- Sample A3 with N_d= **5.3×10^{15} cm^{-3}**: nitridation time of 50 min with angle flow equal to 45°.

In order to determine the thickness of the obtained InN layer and its composition, the nitridation was carried with (XPS) surface analysis using Mg K_α source (1253.6 eV). The structures are tested electrically with a mercury probe used as a temporary gate contact (Section of the gate is S = 7.85 x 10^{-7} m^2). The current–voltage curves were obtained using a HP Semiconductor Parameters Analyser 4155B.

Results and Discussion

Spectroscopy Analysis

Ion bombardment creates metallic indium droplets at the surface of the InP (100) (Akkal, 2000; Sze, 1981). These droplets are consumed by the atomic nitrogen flow coming from a GDS cell to form nitride nanofilm. The In_{4d} core levels obtained for the samples A1, A2 and A3 after nitridation are displayed in Figure. 1, 2 and 3 respectively.

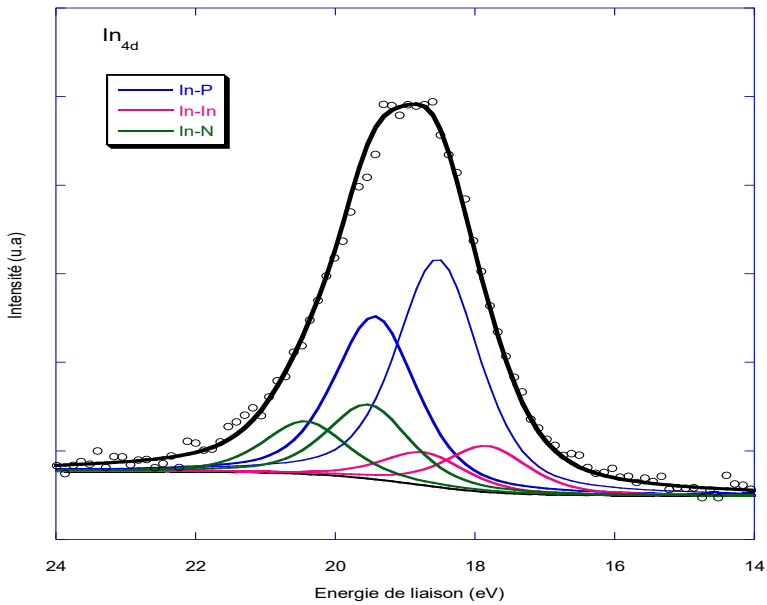

Figure 1. In_{4d} spectra (sample A1)

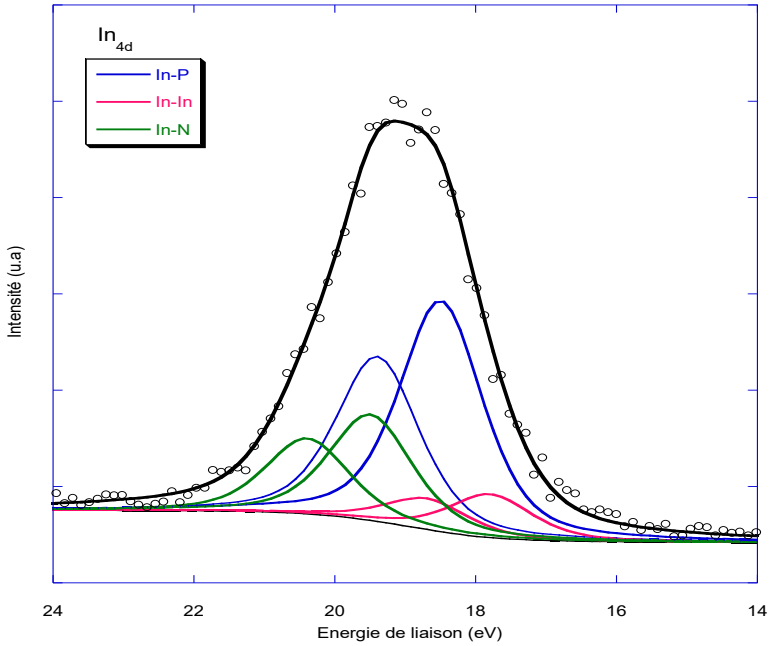

Figure 2. In_{4d} spectra (sample A2)

The In_{4d} core level of the nitridated components can be decomposed into two doublets: one for bulk In-P bonds, the second related to indium nitride as shown in the figures. We note the presence of metallic indium in the In_{4d} core level spectrum showing that the indium clusters were not totally consumed by the nitrogen during the nitridation process. In this case, it is also possible to calculate the theoretical photoelectron intensity. We have considered that the coverage rate of the surface by the nitride layers was equal to unity and that there were two layers of stoichiometric indium nitride. Indeed, indium droplets represent one complete atomic monolayer of metallic indium (θ=25%, height=4ML) (Pan, 1996). So, the maximum number of stoichiometric indium nitride layers which can be formed is two monolayers The spectro were fitted using Shirley background and decomposition into Gaussian and Lorenzian lines shapes. The fitting parameters are summarised in Table 1 (Ambrico, 2005). Moreover, for each environment, we can calculate the theoretical signal coming from the indium atoms.

Table 1. The spectro fitting parameters.

In_{4d}	In-P (bulk)	In-In bonds	In-N bonds
FWHM	1,4±0,2	1,4±0,2	1,4±0,2
% Lorenzian/ Gaussian	50%Gauss, 50% lor	50%Gauss, 50% lor	50%Gauss, 50% lor
Spin-orbit (eV)	0,9	0,9	0,9
Branching ratio	1,3-1,4	1,3-1,4	1,3-1,4
Shift relative to bulk InP	/	-0,7	+1

The intensity for the bulk In-P is written:

$$I_{In-P} = \left(\frac{\alpha_{In}^{h+1}}{1-\alpha_{In}^{2}} \right) i_{In} \tag{1}$$

The intensity coming from In-N is given by the following expression

$$I_{In-N} = 0{,}5(1 + \alpha_{In} + \alpha_{In}^{2} + \ldots + \alpha_{In}^{h-1}) i_{In} \tag{2}$$

h: is the number of the monolayers of InN

I_{In}: is the photoelectron intensity of one atomic monolayer of indium and $\alpha_{In} = \exp\left(-\frac{d}{0{,}96.\lambda_{iIn}}\right)$ the attenuation coefficient of In_{4d} photoelectrons by one monolayer where d is the thickness layer, 0,96: the analyser specific constant and λ_i is the inelastic mean free path of the photoelectrons.

The theoretical ratio I_{InN}/I_{InP} is calculated, it's equal to 0.39 for h=1 and to 0.6 for h=2. The comparison between the experimental and the theoretical ratios allow us to determine the thickness and the number of InN monolayers formed.

Table 2. The results obtained by XPS analysis.

	Sample A1	Sample A2	Sample A3	
Cleaning time (min)	15	15	15	
In-In/In-P	0.35	0.33	0.32	After ionic cleaning
Nitridation time (min)	40	45	50	
Nitrogen flow (angle)	45°	45°	45°	
In-N/In-P	0.32	0.45	0.5	
In-In/In-P	0.202	0.18	0.05	After nitridation

The table 2 summarised the values of experimental ratios obtained for different exposition times at nitrogen flow. We can remark that the amount of the nitride at the surface increase with the exposition time to nitrogen flow. The thickest layer of InN is obtained for a sample A3 wich is nitridated during 50 min and angle flow equal to 45°, with a higher ratio of I_{InN}/I_{InP} (0,5) and lower ratio of $I_{In\text{-}In}/I_{InP}$ (0.03). So the amount of nitride obtained is almost equal to two monolayers of InN (maximum number of stoichiometric indium nitride layers). The sample A1 (nitridated during 40 min) present an experimental ratio equal to 0.32 it corresponds to the theoretical one calculated for one monolayer of indium nitride.

Electrical Analysis

Figure 4 presents the I-V characteristics of the Hg/InN/InP(100) structures for each sample series. The I-V characteristics of these structures show the behavior of rectifying contacts. The conduction is better when the thickness of the InN layer is high. The determination of the electrical parameters was achieved using the characteristic formula expressing thermionic emission current (Benamara, 2006):

$$I = I_S \exp\left(\frac{q(V - R_S.I)}{nkT}\right) \quad (3)$$

$$I_S = S A^{**} T^2 \exp\left(-\frac{q\Phi_{bn}}{kT}\right) \quad (4)$$

Were,

I_s: saturation current;
A**: Richardson constant) (B. Akkal, 2000), S: area of the metal contact (Hg),
T : temperature (300 °K), k: Boltzmann constant, q: electron charge, ϕ_{bn}: barrier height,
n is the ideality factor; it's calculated from the following formula:

$$n = \frac{q}{kT} \cdot \frac{\partial V}{\partial (LnI)} \quad (5)$$

The I-V characteristics show two linear regions, from the first we can determine the ideality factor n, the saturation current I_s and deduce the barrier height ϕ_{bn} . The second region at forward voltage considerably higher

shows existence of serial resistance. In fact, the I-V characteristics reveal a low series resistance (375 Ω) for the samples with large thickness of the InN layer compared with a value of 1513 Ω obtained in the sample with a lower thickness. The saturation current I_s of the samples with two monolayers of InN is the lower. However their potential barrier height is the highest one, so a good Schottky contact is obtained.

Table 3 Electrical parameters calculated from I-V characteristics

	Sample A1	Sample A2	Sample A3
Nitridation time (min)	40	45	50
I_s (A)	2.6×10^{-6}	7.65×10^{-6}	1.92×10^{-6}
N	3.53	3.99	3.07
Φ_{bn} (eV)	0.53	0.512	0.55
R_s (kΩ)	1.513	0.641	0.375
$N_{d(fournisseur)}$ (cm^{-3})	4.6×10^{15}	$4\text{-}7\times10^{16}$	5.3×10^{15}

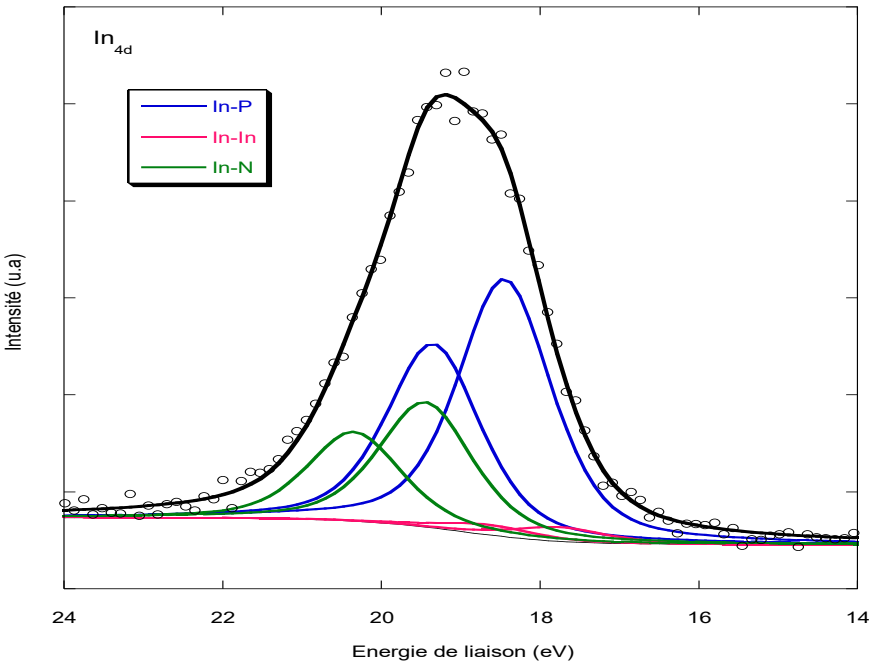

Figure 3. In_{4d} spectra (sample A3).

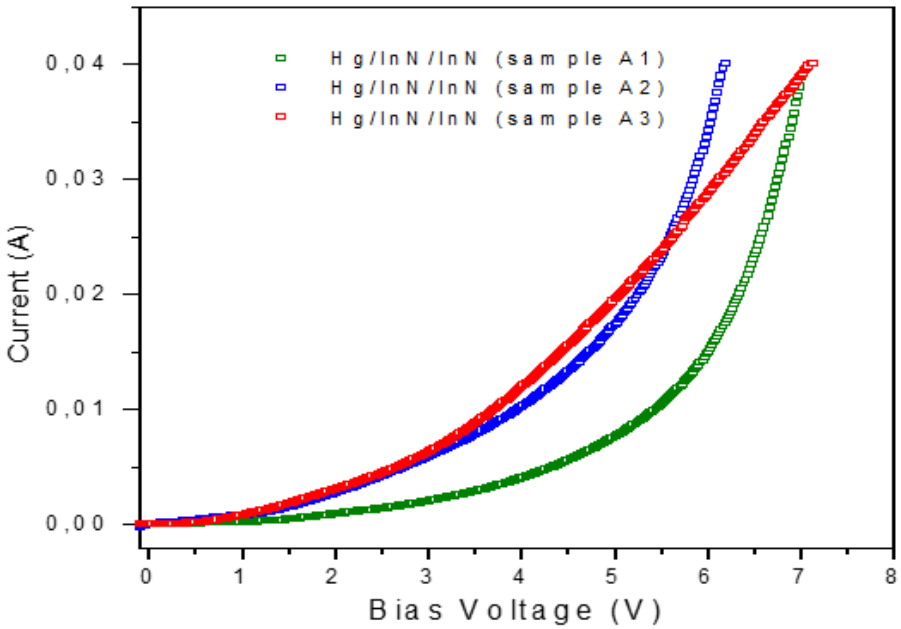

Figure 4. Curent-Voltage characteristics.

Comparing the nitridated samples, the significant value of the ideality factor (n=3.55) is obtained in the nitrided samples with low thickness of InN layer. This effect is probably due to the presence of a tunnel current through the structure. However, the sample with a large thickness presents an interesting ideality factor η= 3 (Because of the consummation In-In at the surface), So this improves the quality of the interface and indicate that interfaces obtained for these structures present a good electronic quality.

Conclusion

The current-voltage characteristics of the Au/InN/InP(100) structures show a behaviour of Schottky According to I–V characteristics, a sufficiently high thickness of the InN in the Hg/InN/InP structures improves the electrical parameters with high barrier heigh and low saturation current.The values of the ideality factor are better for the samples which are well nitrided (total consumption of the metallic indium present on the surface), therefore the InN layer improves the quality of the interface. These results indicate that these devices present a good electronic quality. The electrical results are in good agreement with the spectroscopic ones. They allowed us to highlight the presence of the nitride buffer layer.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest

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