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Optical properties of InAs/InP surface layers formed during the arsenic stabilization process

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Optical characterization has been performed on the InAs/InP surface layer in order to study the arsenic (As) stabilization process of InP. The InAs layers are formed by the simple exchange of phosphorus (P) from the InP surface substrate by arsenic. Samples prepared under arsenic overpressure in the temperature range 450–600 °C have been examined. Luminescence from the InAs layer which acts as a surface quantum well has been detected. We have compared this emission with theoretical calculations and reasonable agreement was observed. Moreover, we have determined that when the arsenic stabilization temperature increases the InAs thickness increases as a consequence of P–As exchange and that after the replacement of the five first top monolayers this exchange seems to stop. We explain this result in terms of the shield effect of the InAs overlayers which efficiently cap the InP substrate against further P evaporation and the restoration of surface structural order which limits any other phosphorus exodiffusion. Comparing our results with those of x-ray photoelectron spectroscopy measurements we have concluded that on samples treated at high temperatures, three-dimensional InAs islands are formed.

I. INTRODUCTION

Conventional molecular beam epitaxy (MBE) consists essentially in condensation of a molecular or atomic flux obtained by evaporation of solid source, on a monocrystalline substrate. The growth of InGaAs and InAlAs by MBE requires a clean and well-ordered InP substrate surface. The main problem is to eliminate a thin native oxide layer without degradation of the surface. Generally, a well-reconstructed InP substrate surface can be obtained by thermal cleaning at ~500 °C under phosphorus¹ (P) or arsenic^{2,3} (As) overpressure, which leads to a P- or an As-stabilized surface, respectively. There is no P source available in most solid source MBE equipments and arsenic stabilization has been shown to be a good procedure to clean InP substrates, preserving both surface morphology and chemical stoichiometry.^{2,3} However, this treatment induces the formation of a thin and pseudomorphic InAs surface layer.⁴ At temperatures of the order of 500–540 °C not only the oxide desorption occurs but also a loss of phosphorus from the surface is induced by thermal desorption.² Under vacuum, In droplets can be formed on the InP substrate surface; however, under As overpressure the desorbed P is immediately replaced by As, forming then the InAs overlayer.^{3,4} Arsenic stabilization has also been used as a passivation process of InP surfaces for the fabrication of metal-insulator-semiconductor (MIS) structures.^{5,6}

The passivation of InP and the MBE growth of semiconductor layers on InP involve a number of technological steps, including substrate pregrowth treatments, which appear to be critical. In this respect there is a need for a detailed knowl-

edge of the physico-chemical and structural properties of the pseudomorphic InAs layer, as a function of the process parameters in relation to its electronic properties. In this framework, we present a study of optical properties of arsenic-stabilized InP(001) surfaces based on low-temperature (5 K) photoluminescence (PL) measurements.

II. SAMPLE PREPARATION

Samples have been prepared in a Riber 2300 MBE reactor. The substrates were InP, (001) oriented, *n*-type nonintentionally doped (10^{16} cm⁻³) wafers of “ready to use” quality from Sumitomo. A thermocouple and an infrared pyrometer were used to measure the sample temperature. The pyrometer was calibrated by directly observing the melting of an InSb crystal mounted on a molybdenum block with In solder. Prior to loading in the MBE chamber, the InP(001) substrates were chemically etched using about 10 cc of an HF-ethanol (1:10) solution using a spinning technique.⁶ Then, the substrates were outgassed in an ultrahigh vacuum chamber at a temperature of 300 °C. The arsenic stabilization was done by heating the InP substrate in the 450–600 °C temperature range under 1.5×10^{-5} Torr of arsenic pressure. The duration of the annealing treatment varied from 2 to 10 min. After the treatment the arsenic shutter was closed when the substrate temperature reached 300 °C. To prevent any remaining arsenic chemisorption, a final annealing treatment was performed under vacuum for each As-stabilized InP sample. The surface morphology and the surface reconstruction of the InP substrates were monitored by 10 keV reflected high-energy electron diffraction (RHEED) and the chemical composition

TABLE I. Characteristics of the various samples studied. T_s is the temperature at which the arsenic stabilization has been done. ML is the number of InAs equivalent monolayers deduced by XPS measurements. t_s is the keeping time to the annealing temperature.

Sample	T_s (°C)	t_s (min)	ML (XPS)	Surface reconstruction
P209 ^d	600	2		4×1
P252	575	0.5	2.60 ± 0.2	4×1
P204	570	2	2.60 ± 0.2	4×1
P253	550	10	1.65 ± 0.2	2×1
P210	500	10	1.50 ± 0.2	2×1
P254	450	10	1.25 ± 0.2	...
P208	450	4	1.25 ± 0.2	...
P207 ^b	525	2		
P203 ^b	500	10		

^dNo XPS measurement has been done.

^bReference samples thermally treated in vacuum without arsenic overpressure.

of the InAs/InP surface layer was probed with high-resolution x-ray photoelectron spectroscopy (XPS). The RHEED and XPS results were already published elsewhere³ and will not be discussed here in detail. The characteristics of the various samples studied are listed in Table I. The average thickness of the InAs layers was determined by XPS. Two InP reference samples were also prepared; they were just annealed under vacuum at two different temperatures. The characteristics of these samples are also given in Table I.

III. RESULTS AND DISCUSSION

In Fig. 1 we present 5 K PL measurements for samples annealed under arsenic at 550, 570, 575, and 600 °C which correspond to an average InAs thickness ranging from 1 to 3 monolayers (ML). At low substrate annealing temperature (T_s) the PL spectrum consists of a main emission with a linewidth of about 150 meV. As T_s is increased, the spectrum broadens being now formed by two or three mixed emissions, and finally, at higher T_s the spectrum consists of a

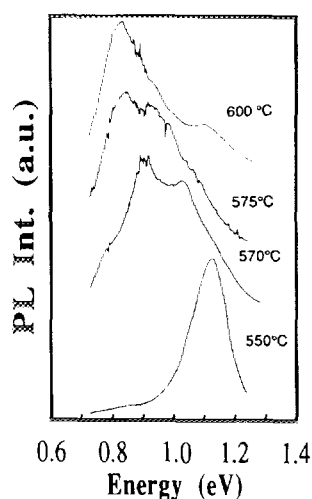


FIG. 1. Low-temperature photoluminescence spectra of arsenic-stabilized samples. The structures around 0.95 eV are due to air absorption.

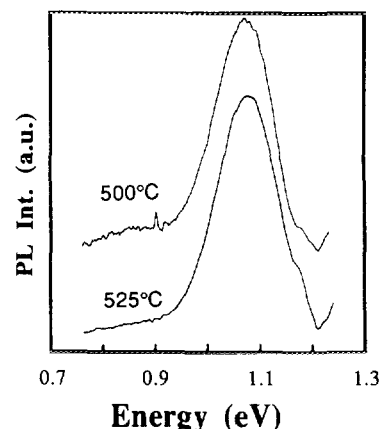


FIG. 2. Low-temperature PL spectra of the InP reference samples, thermally treated in vacuum without As overpressure.

main emission with a shoulder at the high-energy side. We will ascribe (see below) this emission to transitions related to radiative recombinations in the InAs surface layers.

PL results for the InP reference samples, without As treatment, are presented in Fig. 2. A defect band at 1.08 eV is clearly observed and also a less important band at 0.85 eV. At the present time, the precise origin of these two emissions is not completely clear. As a possible origin, Yu has proposed that the 1.08 eV deep level could be related to a complex involving a phosphorus vacancy bound to iron.⁷ However, in our study the substrates are *n*-type nonintentionally doped and the iron concentration should be very small. Temkin *et al.* have proposed for the same level a complex involving a di-vacancy of indium and phosphorus.⁸ For the emission at 0.85 eV, Rao *et al.* have proposed as a possible origin a complex involving a phosphorus vacancy bound to some acceptor impurity.⁹

In Fig. 3, we present PL results for the samples P209, P252, P204, and P253 after chemical etching of the first 50 Å near the surface, including the 1–3 InAs monolayers. This was done in order to check whether or not the emissions reported in Fig. 1 really come from the InAs surface layers. Great differences can be seen when comparing spectra of Fig. 1 (nonetched samples) and Fig. 3. Moreover, after etching identical results to reference samples (Fig. 2) are obtained. This means that after etching samples present transitions just related to deep defect emissions.

From the different results described above, we attribute the PL emissions presented in Fig. 1 as being due to the InAs surface layer. This is based on the following arguments: (i) PL emissions for As-stabilized samples do not correspond in energy to the “defect” bands observed for the reference samples (Fig. 2), (ii) the PL emissions disappear after 50 Å etching (Fig. 3), and (iii) their maximum shifts to lower energies when increasing the arsenic stabilization temperature. We interpret the luminescence of this layer as follow: as InAs has a smaller band gap than InP (0.42 and 1.42 eV at 0 K, respectively),¹⁰ the InAs surface layer can form a quantum well bounded on one side by a monolayer oxide (and vacuum) and on the other side, by the InP substrate.¹¹ Under

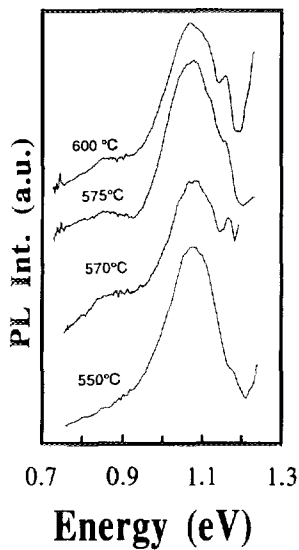


FIG. 3. Low-temperature spectra of As-stabilized samples with about the first 50 Å near the surface chemically etched in order to remove the InAs layers.

these conditions, the photocreated carriers are confined in the InAs well and can thus recombine. For surface quantum wells, the main problem in achieving a high radiative efficiency is the existence of nonradiative transitions involving interface and surface states. Experimentally, light emission is observed thanks to the high density of carriers confined in the quantum well which can saturate these states. The presence of dislocations at the InAs/InP interface is another potential problem which can hinder a high light efficiency. These dislocations can act as carrier traps and kill radiative recombinations. Dislocations are associated to the high degree of lattice mismatch between InP and InAs (3.2%). However, as the InAs average layer thickness (measured by XPS) is less than 3 monolayers, which is smaller than the critical thickness (t_c) for the formation of defects such as dislocations found by Schneider and Wessels ($t_c=5$ ML) for InAs/InP single quantum wells grown by metal-organic vapor phase epitaxy¹² and by Hollinger *et al.* ($t_c=6-7$ ML) for InAs epitaxial layer grown on InP substrate by MBE,¹³ we believe that dislocations (if they exist) are not present in large concentration. So, we associate the observed PL emissions of the Fig. 1 to radiative transitions in the InAs surface quantum well between the fundamental electron (E_1) and heavy hole (H_1) subbands.

In order to determine more precisely the different transitions shown in Fig. 1, we have fitted each spectrum with Gaussian lines. A Gaussian form has been chosen because it well describes an excitonic emission.¹⁴ In quantum wells excitonic line shape has a Gaussian character due to the statistical (Gaussian) distribution of the interface roughness.¹⁵ So, for sample P253 ($T_s=550$ °C), characterized by a 2×1 As-stabilized surface observed by RHEED and an InAs thickness of 1.65 ML deduced by XPS, we have found that the best fit is obtained with a sum of three Gaussians which are ascribed to emissions related to 2 ML (most intense) and 3

TABLE II. Values of energy emission (eV)/linewidth (meV) obtained by fit of the spectra of Fig. 1.

Sample	2 ML	3 ML	4 ML	5 ML	Defect line
P253	1.13/125	1.03/130			0.86/1.72
P204	1.10/205	1.00/150	0.90/95	0.80/110	
P252	1.13/135	1.01/150	0.96/110	0.83/135	
P209			0.94/105	0.83/130	1.08/182

ML of InAs. The third Gaussian at 0.86 eV is ascribed to the deep defect emission as already commented. For sample P204 ($T_s=570$ °C), characterized by a 4×1 In-stabilized surface and an XPS InAs thickness of 2.6 ML, the best fit is obtained with four Gaussians corresponding to emissions of 2, 3, 4 (most intense), and 5 ML. For sample P252 ($T_s=575$ °C) corresponding to an XPS thickness of 2.6 ML and a 4×1 In-stabilized surface, the best fit is also obtained with four Gaussians (2, 3, 4, and 5 ML), the 5 ML one being the most intense. Finally, for sample P209 ($T_s=600$ °C), three Gaussians have been used which correspond to emission of 4 and 5 ML (the 5 ML one being the most intense) and to an emission due to a deep defect emission at 1.08 eV. XPS measurements have not been performed for this last sample. The maximum energy and the full width at half maximum (FWHM) of the Gaussians used in the fits are presented in Table II. It is worth noting that we have not detected defect bands in the majority of arsenic-treated samples due to the weak intensity of those which were hidden by the InAs emission.

In Fig. 4 we show the result of the fit for sample P204. The solid line is the experimental curve, the dashed lines correspond to fitted Gaussians for well thicknesses of 2, 3, 4, and 5 ML, and the dotted line, the sum of these Gaussians. We can see the excellent agreement with the experimental results. The same agreement between experimental and fitted curves was found for the other samples.

Through the above results we have deduced that (i) as the annealing temperature increases the strongest emission line moves to lower energy, meaning that the thickness of the InAs layer increases and that P-As exchange takes place more and more deeply as the temperature increases, (ii) when the surface stabilization changes from As- to In-stabilized

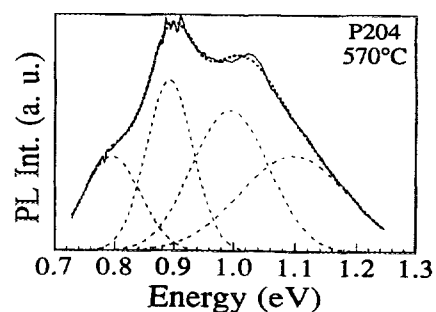


FIG. 4. Fitting result for the PL spectrum of sample P204 resolved into four Gaussian components. Solid line is the experimental result, dashed lines correspond to the fitted Gaussians, and dotted one the sum of these Gaussians.

surface a remarkable change in As penetration depth is observed (from 3 to 5 ML), and (iii) after the replacement of the first five top layers the P–As exchange process seems to stop, as will be discussed below. In Fig. 1, we can observe that for an As-stabilized InP surface prepared at $T_s=550^\circ\text{C}$ the penetration of arsenic atoms in the InP substrate results in the formation of 3 ML InAs layer. When increasing the temperature by 20°C leading to an In-stabilized InP surface, a small quantity of arsenic atoms penetrates up to 5 ML (small hump on the PL spectrum). When increasing now no more than 5°C ($T_s=575^\circ\text{C}$), more and more arsenic atoms penetrate up to 5 ML as can be observed by the increase of PL intensity related to the 5 ML well. At $T_s=600^\circ\text{C}$ (which means an increase of 25°C) the 5 ML related emission is now the main one, but no emission related to well with more than 5 ML thickness is detected. The exchange between As and P seems to be stabilized after the replacement of the five first top layers. We explain this observation in terms of (i) a shield effect of the InAs overlayers which efficiently cap the InP substrate against further P evaporation and (ii) stabilization of the surface by restoration of the structural order which limits the exo-diffusion of phosphorus elements.

XPS studies³ of the As $2p$ and P $2p$ core-level regions have indicated that for samples prepared at temperatures lower than 550°C , arsenic is present within at least the first two top layers while on the sample prepared at $T_s=575^\circ\text{C}$ arsenic is present in at least the first three top layers and probably in the fourth one. However, it was not possible to answer the question whether or not some arsenic atoms have penetrated more deeply in the InP substrate.³ Our results have revealed that for the sample prepared at $T_s=550^\circ\text{C}$ arsenic atoms are not only present in the first two layers but also in the third one (in small concentration). Moreover, for samples prepared at $T_s\geq 575^\circ\text{C}$ we have detected the presence of As atoms in at least the first five layers showing the great sensitivity of low-temperature photoluminescence measurements.

One can note that we have deduced from PL measurements an InAs thickness higher than what was measured by XPS. This difference can be explained as follows: XPS cannot give a direct local measure of the InAs thickness, but gives an evaluation of the number of arsenic atoms on the top layers. This number is obtained by analyzing the relative intensity of the As $3d$ core level peaks.³ The number of InAs monolayers is then deduced by dividing the measured total number of As atoms by the number of As atoms assumed present in one perfect InAs monolayer. Suppose now that the InAs overlayers are not uniform (as occurs, when three-dimensional islands are formed), the number of InAs ML determined by XPS measurements would be underestimated locally because it admits the existence of flat and homogeneous InAs layers. As a consequence, for samples annealed with $T_s\geq 570^\circ\text{C}$ (In stabilized) during 2 min where PL measurements detect the presence of InAs layer with a thickness larger than that measured by XPS, we believe that the InAs layer probably forms three-dimensional islands of height up to 5 ML and lateral extension of at least an exciton radius ($\sim 100\text{ \AA}$). This is in agreement with RHEED measurements which indicate an InAs thickness of $6\text{--}10\text{ \AA}$ ($\sim 2\text{--}3\text{ ML}$) as

being the onset of three-dimensional growth for growth temperatures greater than 525°C .¹⁶ For samples treated at high temperature, Gendry *et al.* argue that from a thermodynamical point of view the strained InAs layers prefer to form islands rather than be flat.¹⁷

For samples prepared at $T_s=450$ and 500°C where XPS measurements give an InAs thickness of 1.25 and 1.5 ML, respectively, PL has just detected the deep defect bands due to thermal degradation of the InP substrate. This means that the PL spectrum of these samples is identical to that of the reference (Fig. 2) and the etched samples (Fig. 3). The non-observation of radiative transitions from InAs in these samples is mainly due to the small thickness of InAs layer, which would not confine enough carriers in the well to saturate the nonradiative transitions related to surface and interface states (as discussed above).

The observed transition energies have been compared to a theoretical calculation of the E_1H_1 transitions for a strained InAs quantum well embedded in InP barriers. We have solved the Schrödinger equation in a finite square well using both envelope function¹⁸ and effective mass approximation and including strain effects on the band structure and on the effective mass. Modification of the band structure due to strain was calculated using the three-band Kane model¹⁹ where the terms of $\epsilon\cdot k$ were neglected.²⁰ The effective mass is given then by the dispersion relation near $k=0$. We have also taken into account the nonparabolicity of the bands. The conduction band offset used was 0.40 eV. We have used this value of ΔE_c because in previous work it has well fitted our results^{11,21} and it also agrees with recent theoretical prevision²² which takes into account the effect of strain on the InAs energy bands. Nevertheless, in the literature a large uncertainty in this value has been reported. Schneider and Wessels¹² have compiled band offsets determined by different groups and techniques and have reported valence-band offset values between 0.27 and 0.48 eV ($\Delta E_c\approx 0.50$ and 0.70 eV). However, for most of the values correction must be done because strain effects on the energy band structure are ignored. The resulting theoretical curve and the experimental points (Table II) are presented in Fig. 5. The general agreement is rather good even using a rectangular form of the potential. This form is not completely adequate to a surface quantum well due to the existence of surface states. These states can pin the Fermi level near the conduction band and bend the potential.²³ Thus, a triangular potential form would better describe our surface quantum wells. Other sources of discrepancy could be (i) noninclusion of exciton binding energy in the calculation, (ii) a possible coupling between the confined states of the quantum wells with both surface and interface states,²⁴ and (iii) the quality of the interfaces, with on the one hand, the possible formation at the interface of an $\text{InAs}_x\text{P}_{1-x}$ graded layer due to some deep penetration of As atoms in the InP substrate, and on the other hand, the chemical and structural disorder of the oxide (vacuum)/InAs interface.

In Table II the values of the maximum energy and the linewidth of the fitted Gaussians are reported. Normally, in this kind of fit for emissions related to the same origins, the maximum energies and the linewidths of the Gaussians

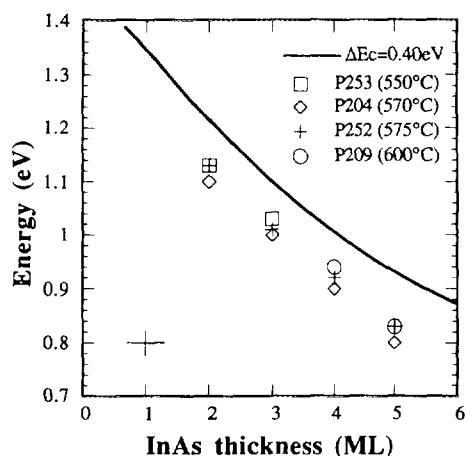


FIG. 5. Dependence of the photoluminescence emission energy with the InAs layer thickness. The solid line is a theoretical calculation for strained InAs/InP square quantum wells using a conduction band offset of 0.40 eV. The cross on the bottom left side represents the error bar.

should be almost constants. We can observe that it happens only for the energy values. For the linewidths despite some fluctuation in the values, an anomalous behavior is observed. The large value of 205 meV obtained for sample P204 for emission related to regions with 2 ML is probably due to an overlapping of this emission with the 1.08 eV deep defect band present in this sample as shown on the etched sample. The same argument can be used to explain the large linewidth of the 5 ML related emissions for samples P252 and P209, but in this latter case overlapping is with the 0.85 eV deep defect band.

The PL linewidth (σ) due to interface roughness, in a square well, is given by²⁵

$$\sigma = \delta \left. \frac{\partial E}{\partial W} \right|_{W_0}, \quad (1)$$

where δ is the two-dimensional island height, E is the energy separation between electron and heavy-hole fundamental levels (E_1H_1), W and W_0 being, respectively, the well size and the average size of the quantum wells. Using the calculation described above for the energy transition E_1H_1 , we have derived, from relation (1) and with an interface roughness described by two-dimensional islands of one monolayer height ($\delta=1$), PL linewidth values of 120 and 68 meV for quantum well thicknesses of 2 and 5 ML, respectively. Comparing these theoretical results with those obtained by the fit (Table II), we can observe that there is not agreement (with exception for the 2 ML emission), meaning that the interface in these samples cannot be described simply with a roughness of 1 ML. This is not surprising if we consider that for InAs layer with thickness greater than three monolayers, three-dimensional islands are formed rather than homogeneous and flat layers.

IV. CONCLUSION

We have performed low-temperature photoluminescence to study the properties of InAs surface layers formed during

the MBE arsenic stabilization process of InP substrates. We have examined samples prepared in the 450–600 °C temperature range. Emissions from the InAs surface layer were clearly identified. These emissions disappear when the InAs surface layer is chemically etched and we only observe emissions from deep defects induced by thermal treatment. We have observed that as the stabilization temperature increases, the InAs layer due to the P–As exchange becomes thicker and thicker with a remarkable change in the As penetration depth when surface stabilization changes for As to In stabilization. The P–As exchange process seems to stop for a thickness of 5 ML. This is a consequence of both the formation of an InAs layer which acts as a shield against further P evaporation and the restoration of the structural order which would limit any other phosphorus exo-diffusion. For In-stabilized surface ($T_s \geq 570$ °C) we have observed PL emissions from InAs layers thicker than those predicted by XPS. This was explained by the formation of three-dimensional islands. In order to better identify the PL transitions, we have fitted our spectra and compared the results with theoretical values. A good agreement has been obtained and the discrepancy observed is mainly due to the complex form of both potential and real interface.

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