



# Influence of buffer layer thickness and epilayer's growth temperature on crystalline quality of $\text{InAs}_{0.6}\text{P}_{0.4}/\text{InP}$ grown by LP-MOCVD

Xia Liu<sup>a,b</sup>, Hang Song<sup>a</sup>, Guoqing Miao<sup>a</sup>, Hong Jiang<sup>a,\*</sup>, Lianzhen Cao<sup>a,b</sup>, Xiaojuan Sun<sup>a</sup>, Dabing Li<sup>a</sup>, Yiren Chen<sup>a</sup>, Zhiming Li<sup>a</sup>

<sup>a</sup> Key Laboratory of Excited State Processes, Changchun Institute of Optics, Fine Mechanics and Physics, Chinese Academy of Sciences, Changchun 130033, PR China

<sup>b</sup> Graduate school of the Chinese Academy of Sciences, Beijing 100039, PR China

## ARTICLE INFO

### Article history:

Received 13 December 2010

Received in revised form

15 March 2011

Accepted 29 March 2011

by X.C. Shen

Available online 6 April 2011

### Keywords:

A.  $\text{InAs}_{0.6}\text{P}_{0.4}$

B. MOCVD

C. Scanning electron microscopy

D. Optical property

## ABSTRACT

$\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers grown on InP (100) substrates using two-step growth method by LP-MOCVD were investigated. A low temperature (450 °C)  $\text{In}_{0.18}\text{Ga}_{0.82}\text{As}$  buffer layer was introduced to relax the lattice mismatch between the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer and the InP substrate. The influence of  $\text{In}_{0.18}\text{Ga}_{0.82}\text{As}$  buffer layer thickness and epilayer's growth temperature on crystalline quality of  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer was characterized by Scanning electron microscopy, X-ray diffraction, Hall measurements, Transmission electron microscopy and Photoluminescence. The experimental results showed that the crystalline quality of  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers could be greatly improved by optimizing the  $\text{In}_{0.82}\text{Ga}_{0.18}\text{As}$  buffer layer thicknesses and the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer's growth temperatures. It was found that, when  $\text{In}_{0.82}\text{Ga}_{0.18}\text{As}$  buffer layer thickness was 100 nm and  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer's growth temperature was 580 °C, the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer exhibited the best crystalline quality and properties.

© 2011 Elsevier Ltd. All rights reserved.

## 1. Introduction

The  $\text{InAs}_y\text{P}_{1-y}$  materials, due to the wide range of attainable band-gap energies from 0.36 to 1.35 eV, are very important in potential applications for the fabrication of laser diodes [1], photodiodes [2], the quantum dot lasers [3], high electron drift velocity devices and quantum well modulators [4], etc. Of all the  $\text{InAs}_y\text{P}_{1-y}$  materials, the  $\text{InAs}_{0.6}\text{P}_{0.4}$  with a high mobility and the band-gap energy of about 0.726 eV is a promising material which could be used in the field of fiber optic communications. It is known that high quality materials are necessary in high performance devices. Unfortunately, due to the lack of a good lattice-matched substrate,  $\text{InAs}_{0.6}\text{P}_{0.4}$  materials were usually grown on lattice-mismatched substrates, such as InP substrates. But the large lattice and thermal mismatches between the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer and the InP substrate lead to the formation of many dislocations and defects in the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers, which deteriorate the crystalline quality of  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers. This problem can be reduced by growth of buffer layers, in which a step-graded buffer layers in composition is often used to inhibit the dislocations to propagate towards the active layer of  $\text{InAs}_y\text{P}_{1-y}$  materials [5–7].

But a single two-step growth method can simplify the growth procedure, in which the low temperature buffer layer is followed and then the growth of epilayer at higher temperatures [8]. However, an  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer with this growth method is rarely reported. An  $\text{In}_{0.82}\text{Ga}_{0.18}\text{As}$  thin film was chosen to act as buffer layer as it could relax the mismatch between the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer and the InP substrate effectively and help to get the high mobility  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers. In the two-step growth method, the growth conditions, such as the buffer layer thickness and the epilayer's growth temperature, are important issues and actively investigated subjects. It is essential to optimize the growth conditions to improve the crystalline quality of  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers.

In this paper, the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers were grown on InP substrates with  $\text{In}_{0.82}\text{Ga}_{0.18}\text{As}$  buffer layers by low pressure metalorganic vapor deposition (LP-MOCVD). The influence of  $\text{In}_{0.82}\text{Ga}_{0.18}\text{As}$  buffer layer thickness and  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer's growth temperature on crystalline quality was investigated. Using the optimum growth conditions, high quality  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayer was obtained and the corresponding reasons of the two respects were also discussed.

## 2. Experiment

All the samples were grown on semi-insulating (100) InP substrates in a horizontal reactor by MOCVD at a pressure

\* Corresponding author. Tel.: +86 431 8462 7073; fax: +86 431 8462 7073.  
E-mail address: [jiangh@ciomp.ac.cn](mailto:jiangh@ciomp.ac.cn) (H. Jiang).

**Table 1**  
Growth parameters and the FWHM of XRC for the samples.

Sample numbers	Buffer layer thickness (nm)	Epilayer's temperature (°C)	FWHM of XRC (arcsec)
A	20	580	1440.0
B	50	580	896.4
C	100	580	554.4
D	200	580	3340.8
E	100	530	1512.0
F	100	550	910.8
G	100	600	2437.2

of 70 Torr. Palladium-diffused hydrogen was used for carrier gas at a total flow of 2.0 L/min. The growth was performed using trimethylindium (TMIn), arsine (AsH<sub>3</sub>) and phosphine (PH<sub>3</sub>) diluted to 10% in H<sub>2</sub> as precursors. The substrates were heated by inductively coupling RF power and temperatures were detected by a thermocouple. The In<sub>0.82</sub>Ga<sub>0.18</sub>As buffer layer growth temperature of all samples with two-step growth method was fixed at 450 °C, and thickness was selected from 20 to 200 nm. However, the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayer's growth temperature was selected from 530 °C to 600 °C, and the thickness was 1.2 μm. Growth parameters for seven samples to be used in this paper were summarized in Table 1. For samples A, B, C and D, the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers were grown at 580 °C, and the In<sub>0.82</sub>Ga<sub>0.18</sub>As buffer layer thicknesses were 20, 50, 100 and 200 nm, respectively. For samples E, F, C and G, the buffer layers with the thickness of 100 nm were grown at 450 °C and the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers were grown at 530, 550, 580 and 600 °C, respectively.

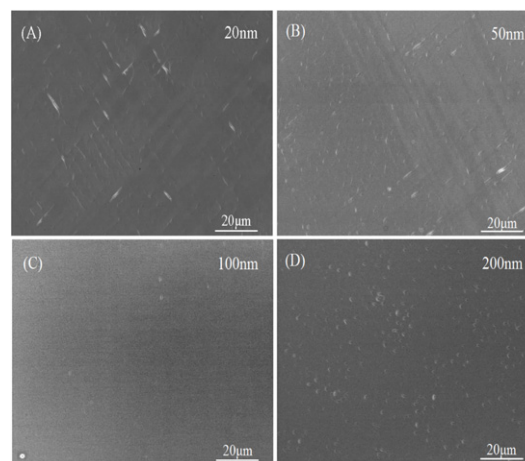
The surface morphology of the epilayers was investigated by scanning electron microscopy (SEM, Hitachi 4800) and atomic force microscope (AFM, Veeco multimode). Crystalline qualities were characterized by double crystal X-ray diffraction (DCXRD, Bruker D8). The carrier concentration and mobility were measured by the Hall measurement (Lakeshore 7707) using the van der Pauw technique at room temperature. The structural properties were investigated by Transmission electron microscopy (JEM 2000 EX) operating at 200 kV. The samples for the TEM measurements were prepared by cutting and polishing to a thickness of approximately 30 μm and then argon-ion milling at liquid-nitrogen temperature to electron transparency. The optical properties were studied by Photoluminescence spectra (PL, Korea) at 77 K in which the 532 nm line was used for the exciting light and the incident light on samples was kept on the same intensity.

### 3. Results and discussion

#### 3.1. Buffer layer thickness on crystalline quality

In order to investigate the buffer layer thickness on the crystal quality of the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers, the rocking curves of the (004) reflection for the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers in the ω mode (XRC) was measured. The FWHM of XRC with different buffer layer thicknesses is also displayed in Table 1. The FWHM values of InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers are 1440.0, 896.4, 554.4 and 3340.8 arcsec, corresponding to the samples A, B, C and D, respectively. This result indicates that the sample with a buffer layer of 100 nm has the minimum FWHM value and the best crystalline quality.

SEM is used to study the surface morphology of the samples. Fig. 1(A)–(D) shows SEM images of samples A, B, C and D, respectively. For sample A, the buffer thickness of 20 nm, a surface with cross-hatched patterns and strip-shaped cracks appears, indicating that the buffer layer is not able to improve the surface morphology of the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayer. For sample B, the buffer thickness of 50 nm, the strip-shaped cracks disappear and some pits appear in the surface. The surface morphology is improved. For sample C, the buffer thickness of 100 nm, the cross-hatched

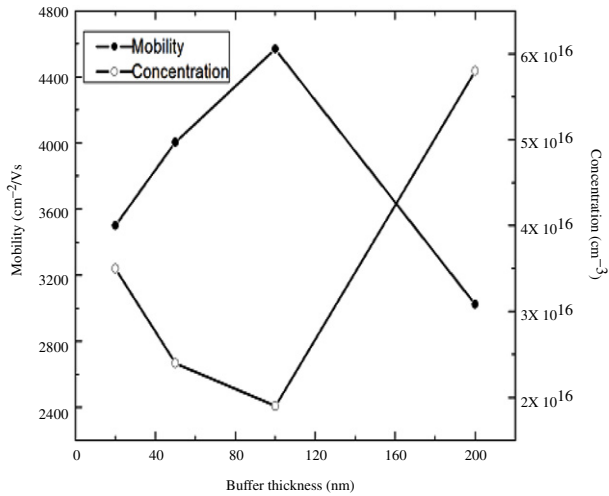


**Fig. 1.** Surface morphology of InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers grown on InP substrates at 580 °C for samples A, B, C and D, respectively.

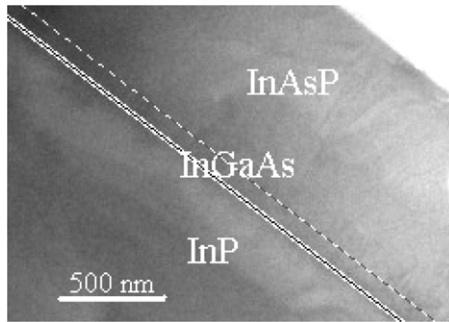
patterns disappear and a very flat surface with very few pits can be visible. It is clear that the surface morphology is greatly improved. For sample D, the buffer thickness of 200 nm, the surface is similar to that of sample C, but a lot of pits appear. It indicates that the surface morphology is degraded. The change of surface morphology can be related to the different buffer layer thicknesses. The buffer layers could not relax the compressive strains between the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayer and the InP substrate completely, and the residual compressive strains still induce kinds of defects such as strip-shaped cracks and pits on the epilayer's surface and misfit dislocations in the epilayers. It is found that a suitable buffer layer thickness can relax the compressive strain in maximum and improved the surface morphology greatly.

Hall measurements were used to characterize the crystalline quality of InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers. The mobility and carrier concentration of InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers for different buffer layer thicknesses are shown in Fig. 2. The mobility of samples increases from 3500.5 to 4568.8 cm<sup>2</sup>/Vs with the buffer thickness from 20 to 100 nm, and it decreases from 4568.8 to 3023.6 cm<sup>2</sup>/Vs with the buffer thickness from 100 to 200 nm. In contrast to the mobility, carrier concentration has the opposite tendency. It is seen that sample C has the highest electron mobility and lowest carrier concentration. As mentioned above, the misfit dislocations introduced by residual compressive strains in the epilayers will act as scattering center and reduce the carrier mobility [9,10]. Therefore, the density of misfit dislocations in the epilayer of sample C is the least and then sample C has the best electrical property.

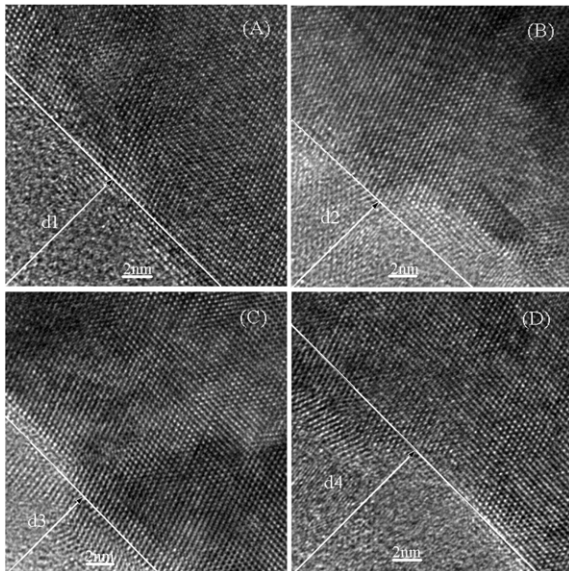
TEM measurements were performed to further study the influence of buffer layer thickness on the quality of InAs<sub>0.6</sub>P<sub>0.4</sub> epilayer. Fig. 3 displays the low-magnification cross-sectional TEM image of InAs<sub>0.6</sub>P<sub>0.4</sub>/In<sub>0.82</sub>Ga<sub>0.18</sub>As/InP layer structure. The interfaces between the InP, InGaAs and InAsP are clearly seen. High resolution Transmission electron microscopy (HRTEM) images of interface between In<sub>0.82</sub>Ga<sub>0.18</sub>As and InAs<sub>0.6</sub>P<sub>0.4</sub> were measured to study the role which the buffer layer plays in reducing the



**Fig. 2.** Carrier concentration and mobility for samples A, B, C and D as a function of buffer layer thickness.

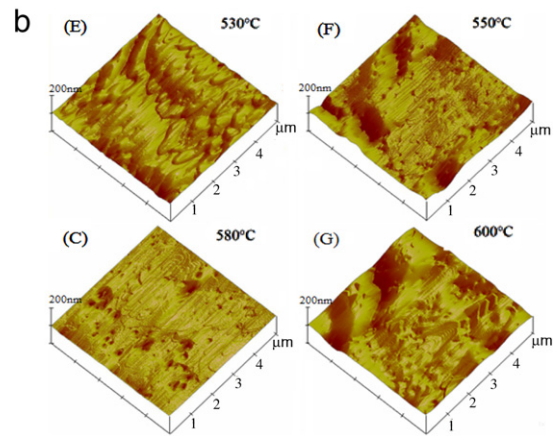
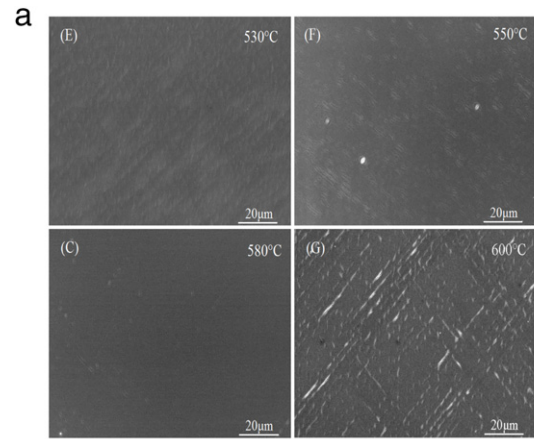


**Fig. 3.** Low-magnification cross-sectional TEM images of the InAs<sub>0.6</sub>P<sub>0.4</sub> layer structure.



**Fig. 4.** Cross-sectional bright-field HRTEM images of (A) sample A, (B) sample B, (C) sample C, and (D) sample D.

dislocations and defects in epilayers. Fig. 4 shows the HRTEM images of interface between the In<sub>0.82</sub>Ga<sub>0.18</sub>As buffer and the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayer of samples A, B, C, and D, respectively. From Fig. 4, white arrows are marked in the images the tail of which points to the interface of In<sub>0.82</sub>Ga<sub>0.18</sub>As and InAs<sub>0.6</sub>P<sub>0.4</sub> and the head of which points to the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayer. It can be seen that the interfaces between the In<sub>0.82</sub>Ga<sub>0.18</sub>As buffer layer and the

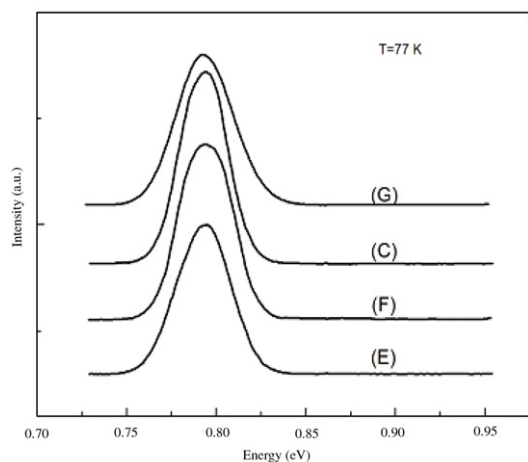


**Fig. 5.** Surface morphology of InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers grown on InP substrates at 580 °C for samples E, F, C and G, respectively. (a) SEM images, (b) AFM images.

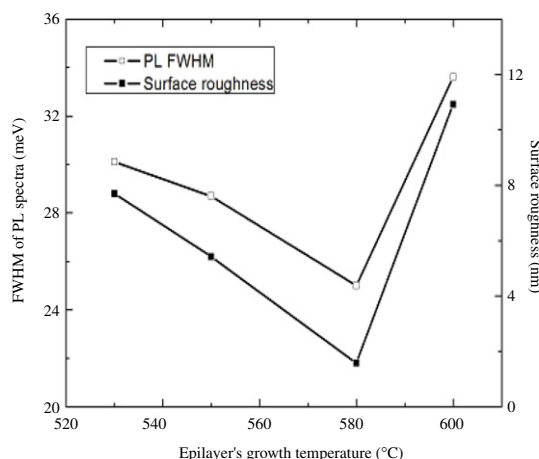
InAs<sub>0.6</sub>P<sub>0.4</sub> epilayer are relatively rough and a number of misfit dislocations and defects [11] in the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayer can be observed. The d1, d2, d3, and d4, are the distances of arrows marked in Fig. 4, which represents the distance of dislocations and defects propagating from the In<sub>0.82</sub>Ga<sub>0.18</sub>As buffer layer to the InAs<sub>0.6</sub>P<sub>0.4</sub> epilayer. It is found that the propagation of dislocations and defects in epilayers are different with various buffer layer thicknesses. The distance d3 has the minimum value and this indicates that a suitable buffer layer thickness can make the dislocations change the direction and bend back to the interface. That is, the density of dislocations and defects of sample C is the least and sample C has the best crystalline quality. Considering the FWHM value of XRD, surface morphology, Hall measurements and TEM measurements, the thickness of the buffer layer which is as thick as 100 nm is the optimum thickness for improving the crystalline quality of InAs<sub>0.6</sub>P<sub>0.4</sub> epilayers.

### 3.2. Epilayer's growth temperature on crystalline quality

Fig. 5(a)–(b) shows SEM images and AFM images of samples E, F, C and G, corresponding to the epilayer's growth temperature of 530, 550, 580 and 600 °C, respectively. It is clear that the sample C grown at 580 °C has the better surface morphology than those of other samples. This change of surface morphology may relate to the epilayer's growth temperature. When the growth temperature was 530 °C, the source materials might not decompose completely and atoms accumulated on the surface, and then the rough grainy surface appeared. This surface morphology can be obviously observed by AFM images shown in Fig. 5(b). When the growth temperature rose to 550 °C, this temperature



**Fig. 6.** Photoluminescence spectra of samples  $\text{InAs}_{0.6}\text{P}_{0.4}$  at 77 K with different growth temperatures of (E) 530 °C, (F) 550 °C, (C) 580 °C and (G) 600 °C, respectively.



**Fig. 7.** The FWHM of PL spectra and root mean square (rms) surface roughness for samples (E), (F), (C) and (G) with different growth temperatures of the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers.

might not provide enough energy for the large grains to coalesce each other. When the growth temperature was 580 °C, the surface atom had enough energy to coalesce each other and a smooth surface morphology appears except for very few pits in the surface. When the growth temperature was higher than 580 °C, such as 600 °C, the surface atom had so much energy to migrate that the three-dimensional growth enhanced and the residual strain was relaxed by introduction of misfit dislocations as the appearance of the strip-shaped cracks and pits [12]. It is found that an optimum growth temperature can improve the surface morphology of the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers. Fig. 5(b) displays the AFM images of  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers with different epilayers' growth temperatures. The root mean square (rms) surface roughness of the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers determined over a ( $5 \times 5 \mu\text{m}^2$ ) area is shown in Fig. 7 as a function of epilayer's growth temperature. The surface roughness of  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers is 7.70, 5.42, 1.58, and 10.92 nm at various growth temperatures. It is evident that the sample grown at 580 °C has the minimum rms value. This result is consistent with that of SEM images.

The FWHM of XRD rocking curves for  $\text{InAs}_{0.6}\text{P}_{0.4}$  (004) plane of samples E, F, C and G, are also shown in Table 1. The values of

FWHM are 1512.0, 910.8, 554.4, and 2437.2 arcsec, respectively. It is seen that the sample grown at 580 °C has the best crystalline quality. The optical properties of the samples are investigated by PL spectra. The 77 K PL spectra of  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers with different growth temperatures are shown in Fig. 6. The band-gap energy of the samples is about 0.795 eV that matches well with that of 0.794 eV calculated by [13]. The FWHM values of PL are 30.1, 28.7, 25.0, and 33.6 meV, as plotted in Fig. 7, corresponding to the growth temperatures of 530, 550, 580 and 600 °C, respectively. It is clear that the sample grown at 580 °C has the minimum value of PL FWHM. This change of PL FWHM can also be related to the growth temperature of epilayers. It is known that some defects and misfit dislocations are known to quench the luminescence and broaden the linewidths of PL spectra [14]. This indicates that sample C has the best crystalline quality. The PL results are in agreement with those of surface morphology and FWHM of XRD. This indicates that there exists an optimum growth temperature for the growth of  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers.

#### 4. Conclusions

In conclusion, we employed two-step growth method to grow the  $\text{InAs}_{0.6}\text{P}_{0.4}$  epilayers on InP substrates using  $\text{In}_{0.82}\text{Ga}_{0.18}\text{As}$  buffer layers by LP-MOCVD. The influence of the  $\text{In}_{0.82}\text{Ga}_{0.18}\text{As}$  buffer layer thickness and epilayer's growth temperature on crystalline quality of the epilayer was studied carefully. It is found that the crystalline quality can be improved greatly by optimizing the buffer layer thickness and epilayer's growth temperature. From the results of SEM, AFM, DCXRD, Hall measurements, TEM and PL spectra, it is found that the optimum thickness of the buffer layer is 100 nm and the optimum epilayer's growth temperature is 580 °C. It is expected that using the simple way may help us to design for the  $\text{InAs}_y\text{P}_{1-y}$  epilayers on InP substrates.

#### Acknowledgment

This work was supported by the National Natural Science Foundation of the China under Grant Nos. 50632060 and 50972141.

#### References

- [1] Y. Imajo, A. Kasukawa, T. Namegaya, T. Kikuta, *Appl. Phys. Lett.* 61 (1992) 2506–2508.
- [2] C.A. Tran, R.A. Masut, J.L. Brenner, R. Leonelli, *J. Cryst. Growth* 124 (1992) 596–603.
- [3] A. Michon, R. Hostein, G. Patriarche, N. Gogneau, G. Beaudoin, A. Beveratos, I.R. Philip, S. Laurent, S. Sauvage, P. Boucaud, I. Sagnes, *J. Appl. Phys.* 104 (2008) 043504.
- [4] R.Y.F. Yip, A.A. Ouali, A. Bensaada, P. Desjardins, M. Beaudoin, L. Isnard, J.L. Brenner, J.F. Currie, R.A. Masut, *J. Appl. Phys.* 84 (1997) 1905–1915.
- [5] M.K. Hudait, Y. Lin, C.L. Andre, P.M. Sinha, C.A. Tivarus, J.P. Pelz, D.M. Wilt, S.A. Ringel, *Appl. Phys. Lett.* 82 (2003) 3212–3214.
- [6] J. Kirch, T. Garrod, S. Kim, J.H. Park, J.C. Shin, L.J. Mawst, T.F. Kuech, X. Song, *J. Cryst. Growth* 312 (2010) 1165–1169.
- [7] S.S. Bui, H.P. Lee, K.M. Yu, *Appl. Phys. Lett.* 90 (2007) 212113.
- [8] H.J. Ko, Y.F. Chen, J.M. Ko, T. Hanada, Z. Zhu, T. Fukuda, T. Yao, *J. Cryst. Growth* 207 (1999) 87–94.
- [9] M.K. Hudait, Y. Lin, C.L. Andre, P.M. Sinha, S.A. Ringel, *Mater. Res. Soc. Symp. Proc.* 722 (2002) K10.2.1–K10.2.6.
- [10] V.W.L. Chin, *J. Phys. Chem. Solids* 53 (1992) 897–904.
- [11] S.P. Ahrenkiel, M.W. Wanlass, J.J. Carapella, L.M. Gedvilas, B.M. Keyes, R.K. Ahrenkiel, H.R. Moutainho, *J. Electron. Mater.* 33 (2004) 185–193.
- [12] X. Liu, H. Jiang, G.Q. Miao, H. Song, L.Z. Cao, Z.M. Li, D.B. Li, *J. Alloys Compd.* 506 (2010) 530–532.
- [13] M. Wada, S. Araki, T. Kudou, T. Umezawa, S. Nakajima, T. Ueda, *Appl. Phys. Lett.* 76 (2000) 2722–2724.
- [14] P. Desjardins, M. Beaudoin, R. Leonelli, G. L'Espérance, R.A. Masut, *J. Appl. Phys.* 80 (1996) 846–852.