High Quality InAlAs on InP for High Sensitivity Photodiodes

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Abstract— The technology of synthesis of InAlAs layers lattice matched to InP by the molecular beam epitaxy technique and the details of pre-epitaxial substrate treatments are considered in this work. We find that with the arsenic beam equivalent pressure of 5×10^{-5} Torr used the optimal structural parameters and luminescent properties were achieved with the substrate temperature to 520°C.

1. INTRODUCTION

The progress in telecommunications requires advances in the technologies of synthesis and processing of multilayer semiconductor heteroepitaxial structures for light emitters and receivers operating in the 1.3–1.6 μm wavelength range. The ultimate obtainable parameters of an optical transmission line are directly dependent on the sensitivity and the frequency range of the photodetector. The technology of creation of high sensitivity avalanche photodiodes has become therefore one of the most important directions of research and development in this field.

To further improve the parameters of high-sensitivity photodetectors for fiber-based transmission lines the utilization of InAlAs/InGaAs heterostructures with an InAlAs avalanche multiplication layer grown lattice-matched on substrates of InP have been proposed [1]. An improvement in the signal-to-noise ratio and a better temperature stability of avalanche multiplication were predicted for the InAlAs-based structures [2, 3]. However, it was shown experimentally that high dark currents and dark count rates, being at least 2–3 orders of magnitude greater than those of the more mature InGaAs/InP diodes, were observed in such structures [1, 4–7]. The high dark count rates were tentatively attributed to the high tunneling currents in the InAlAs layer, possibly via deep levels in the material present due to defects and/or uncontrolled impurities. The realization of the predicted advantages of the InAlAs/InGaAs based structures requires therefore a revision and optimization of the growth technology used for synthesis of detector heterostructures and, first of all, of the aluminum-containing multiplication layer, aimed at minimization of the concentration of centers that cause the interband carrier tunneling.

In this paper we describe the technology of synthesis of layers of InAlAs on (001)InP substrates and detail the pre-epitaxial treatments of the substrates that are required to obtain high quality layers.

2. EXPERIMENTAL TECHNIQUES

The epitaxial structures used in this study were grown on semi-insulating substrates of (001) InP by the molecular beam epitaxial (MBE) technique in a Riber-32P MBE machine. The preliminary chemical preparation of InP substrates included degreasing, removal of a near-surface damaged layer and residual oxidized layer. The samples were degreased in a 1 : 1 solution of monoethanolamine and hydrogene peroxide for two minutes, rinsed in flowing deionized water for 5 min and dried with argon. The substrates were etched using a chemo-mechanical polishing with a specially designed wafer holder. For the removal of the damaged layer, a polishing etchant of 0.3 N solution of $K_2Cr_2O_7$, hydrobromic acid and deionized water in a 2 : 2 : 1 ratio was used [8]. After 5 minutes of polishing (corresponding to an etching depth of 16 μm) the sample was rinsed for 5 min in flowing deionized water and dried with argon. To remove the residual surface oxidized layer the substrates were kept for two minutes in isopropanol saturated with hydrogen chloride and then rinsed several times in pure isopropanol and dried with argon. The resulting morphology of InP substrates was monitored using the atomic force microscopy (AFM) technique in a Solver AFM setup (NT-MDT). The chemical state of the substrate surfaces was studied using the X-ray photoelectron spectroscopy.

Prior to layer growths, the molecular fluxes of constituent materials were measured using an ionization gauge. The substrate temperature was monitored by a single-wavelength infrared pyrometer. The growth process was monitored *in situ* by reflection high-energy electron diffraction (RHEED) technique.

The *ex situ* evaluation of the layer quality was performed by the AFM, X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and photoluminescence (PL) techniques. The XPS spectra were acquired using an ultra-high vacuum Cameca Nanoscan-50 analytical system. The quantitative data on the surface composition of the samples were obtained from measured areas under peaks in the spectra. The corresponding relative sensitivity factors for each element were calculated from the theoretical photoionization cross-sections (Scofield cross-sections) taking into account the transmission function of the spectrometer analyzer. The PL was excited by a cw 532 nm or 405 nm lasers with a power density of $2\,\mathrm{W/cm^2}$. The PL emission was dispersed by a double monochromator equipped with a cooled photomultiplier operated in the photon counting mode. The PL spectra were measured at a temperature of $T = 77\,\mathrm{K}$.

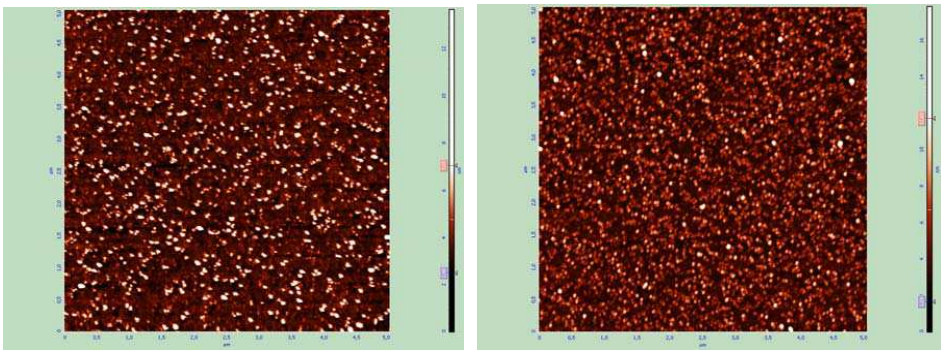
3. EXPERIMENTAL RESULTS AND DUSCUSSION

Figure 1 shows typical AFM images of $5 \times 5\,\mu\mathrm{m}$ regions of InP substrates (a) after the chemical treatment, and (b) after the annealing in the growth chamber in the flux of arsenic (As_4) with a beam equivalent pressure (BEP) of 5×10^{-5} Torr. It is seen that the etched substrate has a smooth surface with the root mean square values of the surface roughness of about 0.28 nm, while after the annealing the roughness increases to 1 nm.

The changes in the substrate surface state during the annealing process were monitored using the RHEED technique. Figure 2 displays RHEED images taken at various temperatures under the arsenic flux with the BEP of 5×10^{-5} Torr. At a substrate temperature of 350°C the (2×3) surface reconstruction is formed, while with temperature increased to 450°C the (2×6) reconstruction appears. At a further increased temperature it gradually disappears, and at 540°C the (4×2) reconstruction is formed. These surface reconstructions are caused by depletion of the surface region of group V elements, P and As. To the best of our knowledge, the appearance of the (2×6) structure is reported for the first time in this work, and is possibly connected with substitution of phosphorus by arsenic atoms. As it is known, under annealing under a flux of P a (2×8) surface reconstruction is observed in the same temperature range [9–11]. At a temperature of 540°C residual oxides are removed, which manifests itself in a change of the ratio of intensities of the peaks and background in the diffraction picture. After the removal of oxides during the cooldown the (2×4) reconstruction appears in the pictures. The subsequent growths of InAlAs layers were performed on substrates annealed at 540°C.

Figure 3 demonstrates the XPS spectra of these substrates. It is seen from the figure that after the annealing in the flux of As_4 at a temperature of 540°C with a subsequent cooling, a large concentration of arsenic is present on the surface. During the annealing process phosphorus atoms desorb from the surface and are partly replaced with atoms of arsenic [12–14], which is accompanied with a formation of islands of InAs. This process leads to the increase in the surface toughness of the substrate. The average lateral size of the InAs islands estimated from AFM maps is about 200 nm.

Layers of $In_xAl_{1-x}As$ with the InAs contents x close to the lattice-matched value of 0.52 and a thickness of 0.5 μm were grown using the technique described above at various substrate temperatures of 480, 500, 515, 520 and 525° in the arsenic flux with a BEP of 5×10^{-5} Torr. According to



(a) (b)

Figure 1: AFM images of 5×5 μm regions of InP substrates (a) after the chemical treatment, and (b) after the annealing in the flux of arsenic.

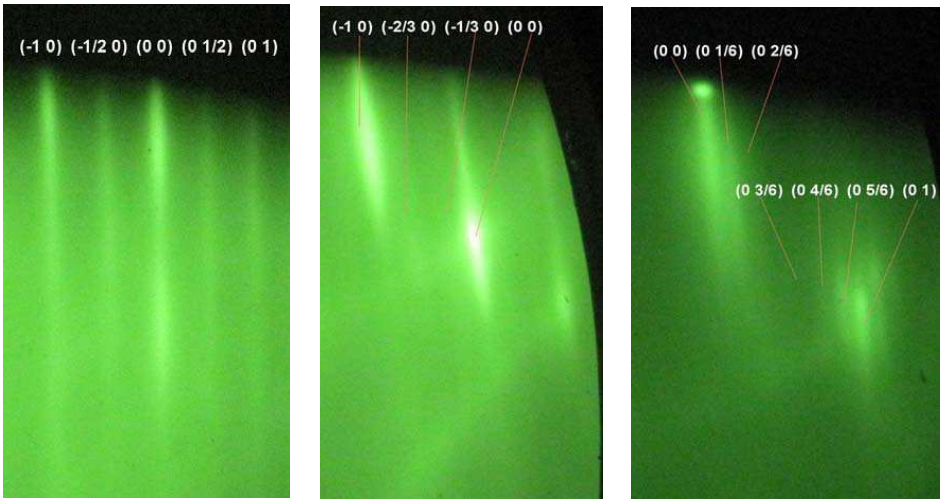


Figure 2: RHEED diffraction pictures of InP substrates during annealing under the arsenic flux with the BEP of 5×10^{-5} Torr at various temperatures: (a), (b) (2×3) reconstruction at a temperature of 350°C taken for azimuthal directions [110] and [−110], respectively,) (2×6) reconstruction at a temperature of 450°.

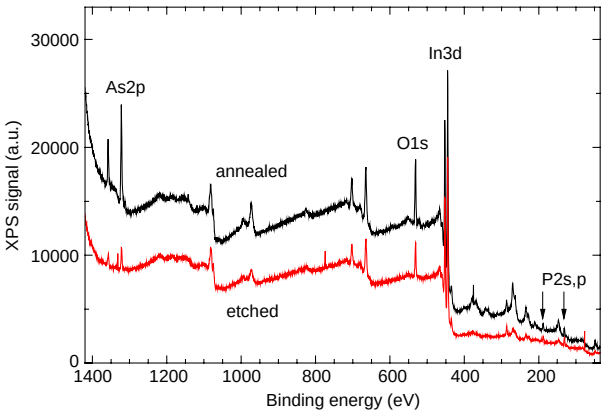


Figure 3: XPS spectra of InP substrates after the chemical treatment (lower curve) and after the annealing at 540°C in the arsenic flux (upper curve).

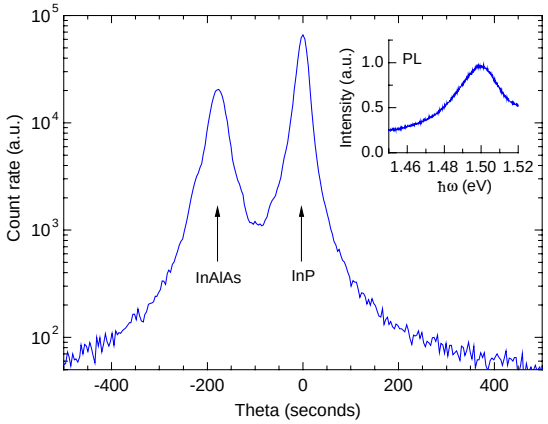


Figure 4: X-ray rocking curve (Cu Kα, 004 reflex) of an $In_{0.53}Al_{0.47}As$ layer grown at a temperature of 520°C. The inset shows the PL spectrum of this layer.

the X-ray diffraction data, the best structural parameters were obtained in layers grown at 520°C. The full width at half maximum (FWHM) of double-crystal X-ray rocking curve was as low as 39″ at this temperature, which was very close to the value of 32″ taken on InP substrate, as illustrated in Figure 4. The inset to the figure shows the PL spectrum of the layer grown at 520°. The spectrum consisted of a single band peaked at 1.499 eV with a FWHM of 27 meV that originated from band-to-band recombination, and in contrast to the samples grown at lower temperatures no impurity-related bands have been observed.

4. CONCLUSION

The conditions of pre-epitaxial chemical treatment and vacuum annealing of (001) InP substrates and MBE growth InAsAs layers were considered in this paper. We found that with the arsenic beam equivalent pressure of 5×10^{-5} Torr used in this work the optimal structural parameters and luminescent properties were achieved with the substrate temperature to 520°C.

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