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Reciprocal lattice mapping of InGaAs layers grown on InP(001) and GaAs(001) substrates♣

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The structure of surfaces of InGaAs(InAs) layers grown on InP(001) and GaAs(001) by molecular beam epitaxy (MBE) was studied by high-resolution X-ray diffractometry. The reciprocal lattice mapping and the rocking curve technique were used to determine distribution of misfit dislocations in the layers. Directional dependence of dislocation density in InGaAs strained layers grown at two-dimensional (2D) growth mode was observed. It was found that anisotropic distribution of dislocations in the InGaAs layers resulted from development via bending in the interface plane of dislocations present in the InP substrate. Simultaneously, homogeneous distribution of dislocations in relaxed InAs layers, grown on InP as well as GaAs substrates, has been detected. At the initial stage these epitaxial layers were grown due to tree-dimensional (3D) island mode. The reciprocal lattice maps confirm that coalescence of islands during the epitaxy generates dislocations that in turn homogeneously distribute in the layer. It seems that the growth mode rather than lattice mismatch determines density of dislocations in InAs epitaxial layers grown on InP and GaAs substrates. However, lattice mismatch influences relaxation process in lattice-mismatched layers. Transport properties of relaxed InAs layers strongly depend on growth temperature.

Keywords: InGaAs, molecular beam epitaxy, X-ray diffraction, diffuse scattering

1. Introduction

InGaAs (InAs) alloys have been the subjects of intense studies over the past few years, due to their importance as opto-electronic materials. Except the alloy with composition $In_{0.53}Ga_{0.47}As$, which is lattice-matched to InP, all other compositions are usually grown epitaxially on GaAs or InP substrates, and they are therefore "mismatched materials". It is important to realise that in this case the lattice mismatch between epitaxial layer and substrate is quite big. This difference has a maximum value of $\approx 7.2\%$ between InAs and GaAs and 3.2% between InAs and InP. Therefore, growth of thick layers and structures is accompanied by formation of misfit dislocations. The lattice mismatch present at the heterointerface is relaxed into defects through the creation of dislocations in epilayers after the growth of critical thickness. In spite of those differences in lattice constants of epitaxial layers and substrates, modern technologies allow to grow good quality materials. It is generally believed that misfit dislocations substantially degrade transport properties [1]. Nevertheless, it was recently demonstrated that non-inverted InGaAs/AlGaAs HEMT devices with the best device and circuit performance contain a linear array of misfit dislocations [2] and the transistor performance is degraded only when orthogonal array of misfit dislocations forms. On the other hand, studies have shown that the dislocation density in InAs epilayers on GaAs and InP increases with decreasing thickness and most of the defects are confined in the first 0.2 μm of epilayer [3–5].

Recently, triple-axis diffraction technique (TAD) has been developed into powerful tool for studying misfit dislocations in semiconductor layers [6] and in heterostructures [2]. It has been found that observa-

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tion of directional diffuse scattering provides a more sensitive means of detecting misfit segments than other commonly used techniques [2].

Here we present results of directional X-ray diffraction studies of InGaAs (InAs) layers with different lattice mismatch with respect to the substrate. Azimuthal oriented diffuse scattering, obtained by means of TAD technique, has been observed on the reciprocal lattice maps. The maps registered together with X-ray topographs allowed characterising misfit dislocations existing in the structures. The strain state and dislocation densities in the layers have been determined.

2. Experimental details

InGaAs and InAs epilayers were grown by the conventional solid-source MBE in a Riber 32P system. The experiments were performed on the following structures:

- nearly lattice-matched $In_{0.525}Ga_{0.475}As$ layers grown on InP (001) substrates (labelled #240.97),
- moderately lattice-mismatched InAs layers on InP (001) substrates (labelled #309.98) and strongly lattice-mismatched InAs layers on GaAs(001) substrates (labelled #292.98 and #302.98),
- strongly lattice-mismatched InAs p–i–n structures on GaAs (labelled #304.98).

$In_{0.525}Ga_{0.475}As$ 2–μm thick layer after oxide desorption at 510°C was grown directly on InP SI substrate. The epilayer was grown at (2×4) reconstructed surface with the growth rate 1 μm/h at the substrate temperature 520°C. In the studies, tetrameric As_4 was used and flux ratio $\varphi(As_4)/\varphi(Ga + In) \approx 7$ was maintained.

InAs 4–5 μm thick layers were grown on InP SI or on GaAs SI substrates. The only difference was that layers grown on GaAs were proceeded by additional 1 μm thick GaAs buffer layer grown at substrate temperature $T_s = 580°C$. During the growth of InAs, a clear diffuse (1×1) reflection high-energy electron diffraction (RHEED) pattern was observed and flux ratio $\varphi(As_4)/\varphi(In) \approx 4$ was maintained. The layers were grown at three substrate temperatures $T_s = 450°C$ – #292.98, $T_s = 480°C$ – #309.98 and $T_s = 500°C$ – #302.98.

Additionally p–i–n InAs/GaAs detector structure was grown at $T_s = 500°C$ (#304.98). In the structure p– and n– type layers were doped with Be (1×10^{18} cm^{-3}) and Si (1×10^{18} cm^{-3}), respectively. Device structure was grown on a GaAs buffer layer deposited on semi-insulating GaAs (001) substrate. It consisted of an n–type InAs layer 1.5 μm thick, followed by an 3.6 μm undoped InAs layer and a p–type InAs layer 1 μm thick. After growth, the $In_{0.525}Ga_{0.475}As$/InP, InAs/InP and InAs/GaAs layers were characterised *ex situ* by Hall effect measurements at both room and liquid nitrogen temperatures. Typical van der Pauw samples with annealed In ohmic contacts were used.

Microstructure of epilayers was examined by high-resolution X-ray diffractometry in double- and triple-axis configuration. In order to determine the strain status of the layers, in-plane ($a_{\parallel}$) and out-of-plane ($a_{\perp}$) lattice parameters were measured and the symmetrical and asymmetrical reciprocal lattice maps were recorded. The lattice parameters were determined by the Bond method or by the method of the direct measurements of the Bragg angle [7] using the symmetrical 004 and asymmetrical 117, 115 and 335 reflections. The $CuK_{\alpha1}$ radiation was used in the experiment. The strain-state of the samples is described by the strain parameter γ, where γ is defined by

$$1 - \gamma = (a_{\parallel} - a_s) / (a_{relax} - a_s) \qquad (1)$$

The symbols $a_{\parallel}$ and a_{relax} denote the in-plane lattice parameter and the relaxed one of the layer material, respectively; a_s is the lattice parameter of the substrate. Therefore, $\gamma = 1$ corresponds to a fully strained structure, whereas $\gamma = 0$ corresponds to a full relaxation.

X-ray reciprocal lattice maps were recorded by performing a series of $2\Theta - \omega$ scans. Investigation was focused on directional character of both full width at half maximum (FWHM) of the (004) rocking curve and diffuse X-rays scattering observed on reciprocal lattice maps. Therefore, the measurements of FWHM of the (004) rocking curves as well as reciprocal space maps were made for two positions of each sample, in which the [110] and [-110] directions were perpendicular to the diffraction plane. The crystallographic direction in-plane was determined from the shape of etch figures revealed in chemical treatment. In order to visualise dislocation distribution in the layers X-ray reflection topographs were made. Due to the fact that it is difficult to calculate the density of dislocations from the topograph, (individual dislocations are not resolved), it was calculated from the rocking curves broadening for several hkl reflections [8].

3. Results and discussion

Results of Hall effect and electrical resistivity measurements exhibited n–type conduction of

Table 1. Electrical properties of InGaAs and InAs epitaxial layers on InP or GaAs substrates grown by MBE.

Sample	T_s (°C)	Layer thickness (μm)	n (300 K) (cm^{-3})	μ (300 K) (cm^2/Vs)	n (80 K) (cm^{-3})	μ (80 K) (cm^2/Vs)
#240.97 (InGaAs/InP)	520	2.0	4.0×10^{15}	4060	7.0×10^{14}	5780
#309.98 (InAs/InP)	480	5.0	6.7×10^{16}	8200	5.8×10^{16}	11600
#292.98 (InAs/GaAs)	450	4.1	5.1×10^{17}	6900	4.8×10^{16}	7300
#302.98 (InAs/GaAs)	500	4.1	1.1×10^{16}	12900	6.6×10^{15}	25800

undoped samples. The values of the determined carrier concentration n and the mobilities μ are collected in Table 1. In order to reduce influence of layer thickness on transport properties the measurements have been performed on relatively thick epilayers.

It has been detected that free carrier concentration in the nearly lattice-matched $In_{0.525}Ga_{0.475}As$/InP layer (#240.97) was about one order of magnitude lower than that found in the best strongly lattice-mismatched InAs/GaAs layer (#302.98). Significant differences in structural properties of these layers, like strain relaxation and dislocation densities, have been observed and are presented in Table 2.

It can be noticed from the Table 2 that $In_{0.525}Ga_{0.475}As$ layer, grown on InP substrate (#240.97) at two-dimensional (2D) growth mode, was nearly completely strained ($\gamma = 0.99$), as determined from rocking curve measurements. On the other hand asymmetrical reciprocal lattice maps presented in Fig. 1 have confirmed this. Identical values of x co-ordinates of reciprocal points of the layer and the substrate also confirm existence of strain in the layer. Additionally, measurable diffuse scattering is clearly observed in the maps. Although extensive diffuse scattering exists in both {110} directions, the extent of it differs significantly for each azimuthal orientation. Such effect is always observed for the samples exhibiting non-homogeneous, orientation-dependent dislocation density. Indeed the X-ray topography confirmed that dislocations were present, although with different densities, in the two directions as shown in Fig. 2. However, the average dislocation density in the layer was lower than 10^5 cm^{-2}, as required to induce detectable changes of the FWHM of rocking curve [10]. Orientation dependent diffuse scattering seems to be not only confined to $In_{0.525}Ga_{0.475}As$ epitaxial layer since similar behaviour has also been observed in InP substrate, as it is shown in Fig. 3. The presented result can be explained in the following way. Misfit dislocations in the InGaAs/InP system develop most probably via bending in the interfaceplane of the dislocations present in the substrate, i.e. by mechanism proposed by Matthews et al. [9]. The higher density of [-110] dislocations in InP substrate and their higher mobility resulted in anisotropy of dislocation density appearing at the interface. Above result is similar to that observed for AlGaAs on GaAs by Domagała et al. [10] and for InGaAs on GaAs by Kavanagh et al. [11]. Therefore, it seems that in III–V layers having zincblende structure [-110] dislocations have a higher velocity than [110] ones. This behaviour seems to be common for epitaxial layers grown on oriented substrates. In the case of epitaxial layers grown on misoriented substrates the opposite results were reported [12].

In contrast to previously discussed InGaAs/InP layer all InAs samples (layers and p-i-n structures) grown on InP as well as on GaAs substrates were re-

Table 2. Structural properties of of InGaAs and InAs epitaxial layers on InP or GaAs substrates grown by MBE.

Sample	$a_\perp$ (Å)	$a_\parallel$ (Å)	γ	FWHM [110] (arc sec)	FWHM [-110] (arc sec)	Dislocation density (cm^{-2})
#240.97 (InGaAs/InP)	5.8629	5.8700	0.99	20	22	9×10^4
#309.98 (InAs/InP)	6.1259	6.0218	0.2	167	171	5×10^7
#292.98 (InAs/GaAs)	6.0623	6.0557	0.008	232	238	1×10^8
#302.98 (InAs/GaAs)	6.0618	6.0559	0.006	350	360	3×10^8
#304.98 (p-i-n InAs/GaAs)	6.0613	6.0569	0.003	178	184	6×10^7

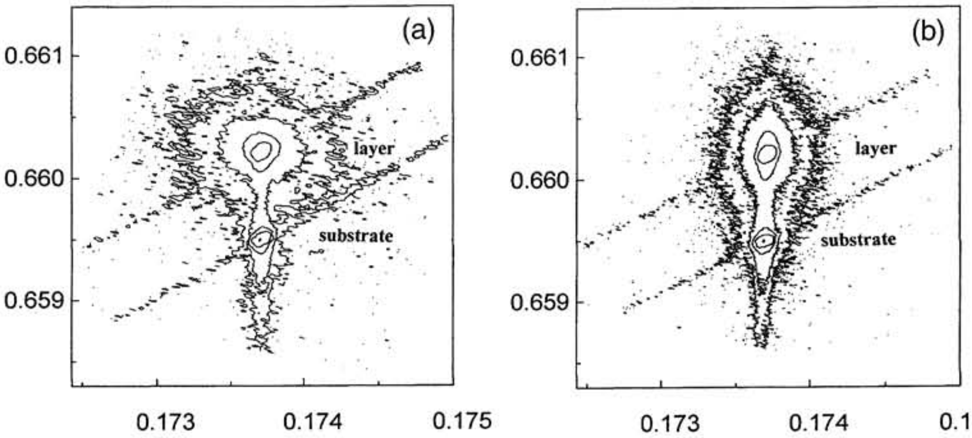

Fig. 1. (115) reciprocal lattice maps of the InGaAs/InP (#240.98) structure: (a) sample aligned with [-110] direction perpendicular to the diffraction plane, (b) sample aligned with [110] direction perpendicular to the diffraction plane. The axis are marked in λ/2d units, where λ is wavelength and d is inter-planar distance. The ratio of neighbouring contours is equal to 10. The diffuse scattering is greater when the diffraction plane is perpendicular to [-110] (a) than to [110] (b).

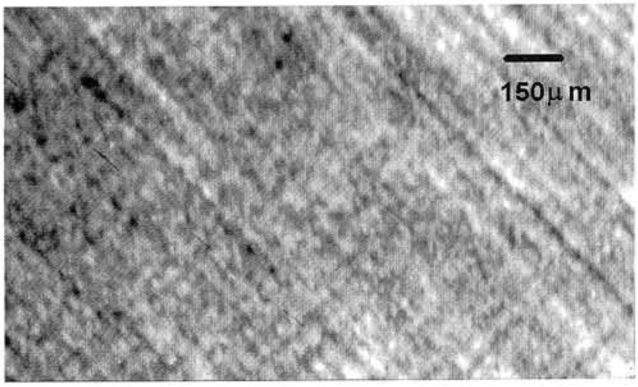

Fig. 2. (044) X-ray topograph of the $In_{0.525}Ga_{0.475}As$ layer on InP substrate (#240.97).

laxed, see Table 2. However, residual strain, lattice-mismatch dependent, has been found. The InAs layer grown on InP substrate (#309.98) was only partially relaxed, when difference of lattice constants was 3.2%. In this case strain parameter $\gamma = 0.2$ was determined. On the other hand the InAs layers grown on GaAs substrates (#302.98) were nearly completely relaxed. In this case lattice mismatch was about 7.2%. For all InAs/GaAs layers strain parameter was similar and the small value of $\gamma < 0.01$ have always been detected.

Table 2 shows also the values of FWHM of the (004) rocking curves of the layers and p-i-n structure measured for the samples alignment in which the [-110] and [110] directions were perpendicular to the

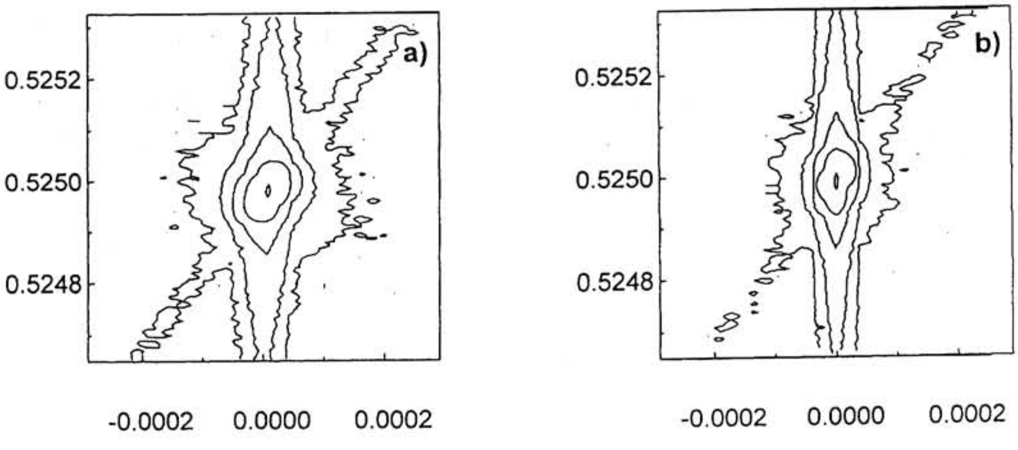

Fig. 3. (004) reciprocal lattice maps of the InP substrate: (a) sample aligned with [-110] direction perpendicular to the diffraction plane, (b) sample aligned with [110] direction perpendicular to the diffraction plane. The axes are marked in λ/2d units, where λ is wavelength and d is inter-planar distance. The ratio of neighbouring contours is equal to 10. The diffuse scattering is greater when the diffraction plane is perpendicular to [-110] (a) than to [110] (b).

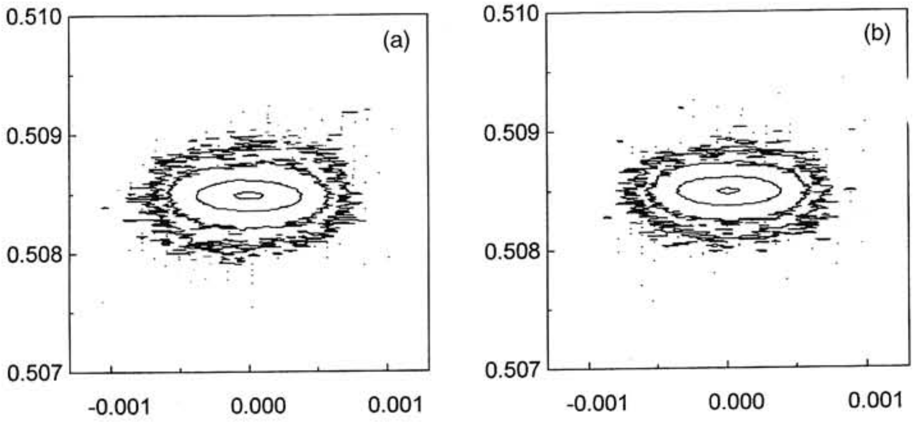

Fig. 4. (004) reciprocal lattice maps for p–i–n InAs structure on GaAs substrate (#304.98): (a) sample aligned with [-110] direction perpendicular to the diffraction plane, (b) sample aligned with [110] direction perpendicular to the diffraction plane. The axis are marked in λ/2d units. The ratio of neighbouring contours is equal to 10. The diffuse scattering is similar in both positions.

diffraction plane. It has been found that for all InAs samples the values of the FWHM were below 360 arcsec and were slightly directional dependent. The main factor influencing the rocking curve is often high dislocation density. Indeed direct correspondence of the FWHM and average dislocation density has been observed. The lowest value of the FWHM (< 170 arcsec) has been determined in the InAs layer containing dislocations at the density about 5×10^7 cm^{-2}.

In order to confirm principal source of relatively large values of FWHM in InAs layers, reciprocal lattice mapping has been performed. Similar maps have been observed for all InAs layers and typical ones obtained for p-i-n detector structure are presented in Fig. 4. In this case due to the high difference of lattice constants of substrate and layer the reciprocal point of layer is only presented. As it can be seen in Fig. 4 the source of rather large FWHM in investigated layers is, in fact, the mosaicity. It is worth to notice that extensive diffuse scattering exists in both {110} directions. It means that density of dislocations in the both directions is comparable and is relatively high. This results from the three-dimensional (3D) mode of growth of InAs layers [3,4]. The critical thickness of InAs layer grown on GaAs or InP substrate is limited to only few monolayers, so that while the epitaxial growth starts with 2D mode growth. The structure of the layer is soon transformed into a high density of small 3D islands to reduce the surface energy due to the lattice mismatch. The islands grow generating a high density of dislocations, and the strain due to the lattice mismatch is released within the initial stage of island growth. The coalescence of islands results in heavy interaction of the misfit dislocations at each merged island boundary and the threading dislocations are generated. Most of the threading dislocations annihilate within a thickness of 0.2 µm from the interface. After completion of island merging, 2D growth continues and finally thick InAs having smooth surfaces are obtained [3,4].

In spite of that similar densities of misfit dislocations in InAs layers have been found (Table 2) and their transport properties differ significantly. Independently on the kind of substrate material (InP or GaAs) lower free carrier concentration and higher carrier mobility were detected in the layers grown at the higher substrate temperature T_s (Table 1). This may suggest that mismatch dislocations observed in InAs are not the main reason of degradation of electrical properties of MBE grown layers. This behaviour confirms that detected misfit dislocations are confined in relatively narrow region just at the heterointerface and relatively small amount of threading dislocation exists. Therefore such layers have acceptable quality to be applied in device structures when relatively thick active layer is needed. It has been proved that infrared detectors fabricated from p–i–n structure (#304.98) discussed here, exhibited electrical characteristics only slightly degraded as compared to calculated ones [13].

4. Conclusions

Diffuse scattering of InGaAs/InP, InAs/InP and InAs/GaAs layers with different lattice-mismatch with respect to the substrates has been investigated. It has been detected that diffuse scattering depends strongly on sample orientation only for the strained layers. Correlation of diffuse scattering with the array of dislocation in X-ray topographs has been observed. Experimental results show that in undoped InGaAs layer grown on (001) oriented InP substrate [-110] dislocations have a higher velocity than [110] ones. Anisotropic distribution of dislocations in the layer results from development via bending in the heterointerface of dislocations present in the InP substrate. This behaviour seems to be typical for III–V epitaxial layers having zincblende structure grown on oriented substrates.

Simultaneously, isotropic distribution of dislocations in relaxed InAs layers has been observed. It has been found that misfit dislocations are not main reason of transport properties of MBE grown InAs layers. In spite of relatively high density of misfit dislocations detected InAs layers exhibit acceptable properties to be used in device applications. This results from confinement of misfit dislocations to the narrow region at the heterointerface. It seems that growth mode rather than lattice mismatch determines density of dislocations in InAs epitaxial layers grown on InP and GaAs substrates. However, lattice mismatch influences relaxation of InAs layer.

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References

1. L.D. Nguyen, D.C. Radulescu, M.C. Foisy, P.J. Tasker, and L.F. Eastman, "Influence of quanum-well width on device performance of $Al_{0.30}Ga_{0.70}As/In_{0.25}Ga_{0.75}As$ (on GaAs) MODFET's", *IEEE Trans. Electron Devices* **36**, 833–837 (1989).
2. M.S. Goorsky, M. Meskinpour, D.C. Streit, and T.R. Block, "Diffuse X-ray scattering from misfit dislocations at semiconductor hetero-interfaces", *J. Phys. D: Appl. Phys.* **28**, A92–A96 (1995).
3. S. Chang, T. Chang, and S. Lee, "The growth of highly mismatched $In_xGa_{1-x}As$ ($0.28 < x < 1$) on GaAs by molecular beam epitaxy", *J. Appl. Phys.* **73**, 4916–4926 (1993).
4. K. Otamoto, R. Hananoki, and K. Sakiyama, "InGaAs epilayers of high In composition grown on GaAs substrates by molecular beam epitaxy", *Jpn. J. Appl. Phys.* **33**, 28–32 (1994).
5. S. Chang, S. Lee, C.R Chen, and L.J. Chen, "Dislocation generation mechanism of $In_xGa_{1-x}As$ ($0 < x < 1$) epilayers grown on (100) InP substrates by molecular beam epitaxy", *J. Appl. Phys.* **75**, 1511–1516 (1994).
6. H. Heinke, M.O. Moller, D. Hommel, and G. Landwehr, "Relaxation and mosaicity profiles in epitaxial layers studied by high resolution X-ray diffraction", *J. Cryst. Growth* **135**, 41–52 (1994).
7. P.F. Fewster and N.L. Andrew, "Absolute lattice-parameter measurement", *J. Appl. Cryst.* **28**, 451–458 (1995).
8. E. Ayers, "The measurements of threading dislocation densities in semiconductor crystals by X-ray diffraction", *J. Cryst. Growth* **135**, 71–76 (1994).
9. J.W. Matthews, S. Mader, and T.B. Light, "Accomodation of misfit across the interface between crystals of semiconducting elements or compounds", *J. Appl. Phys.* **41**, 3800–3804 (1970).
10. J. Domagała, J. Bąk-Misiuk, J. Adamczewska, Z.R. Zytkiewicz, E. Dynowska, J. Trela, E. Janik, D. Dobosz, and M. Leszczyński, "Anisotropic misfit strain relaxation in thin epitaxial layers", *Phys. Stat Sol. A* (in publish).
11. K.L. Kavanagh, M.A. Capano, L.W. Hobbs, J.C. Barbour, P.M. Maree W. Schaff, J.W. Mayer, D. Pettit, J.M. Woodall, J.A. Stroscio, and R.M. Feenstra, "Asymmetries in dislocation densities, surface morphology, and strain of GaInAs/GaAs single heterolayers", *J. Appl. Phys.* **64**, 4843–4852 (1988).
12. P. Werner, N.D. Zakharov, Y. Chen, Z. Liliental-Weber, J. Washburn, J.F. Klem, and J.Y. Tsao, "Stress releasing mechanisms in $In_{0.2}Ga_{0.8}As$ layers grown on misoriented [001] substrate", *Appl. Phys. Lett.* **62**, 2798–2800 (1993).
13. J. Piotrowski and J. Kaniewski, "Optimisation of InGaAs infrared photovoltaic detectors", *IEE Proc. Optoelectronics* (in publish).