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Defect structure changes in thin layers of semiconductors annealed under hydrostatic pressure

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The effect of annealing under enhanced hydrostatic argon pressure (1.2 GPa) on the defect structure of thin GaAs:Be, InAs and AlGaAs and on the layers grown on GaAs substrates was investigated by X-ray diffraction methods. The strain state of the homoepitaxial GaAs:Be layers remained unchanged after the high pressure–high temperature (HP–HT) treatment, but additional defects were created on primarily existing structural irregularities. The treatment of $A^{III}B^{V}$ heterostructures resulted in the changed strain state and dislocation density, which are dependent on the bulk modulus of the layer and substrate materials.

1. Introduction

As it follows from recent investigations of the high pressure–high temperature (HP–HT) effect on strained hydrogen- or oxygen-implanted silicon, Si:H and Si:O, [1]–[3] and on the thin AlGaAs layers deposited on the GaAs substrate (*e.g.*, [4], [5]), the HP–HT treatment can result in the changed defect structure and strain state of such samples.

An influence of the HP–HT treatment on the interface of the layered system as dependent on the primary defect structure and primary strain of the layers was determined in the present paper using the high resolution X-ray diffractometry. In what follows the experimental results for the homoepitaxial GaAs:Be/GaAs and heteroepitaxial InAs/GaAs, AlGaAs/GaAs samples are presented.

The influence of the HP–HT treatment on the semiconductor layer structure is related to the strain generated during the treatment due to differences in the thermal expansion α and the bulk modulus B of the layer and substrate materials.

The mismatch-related strain (ε_{HP-HT}) at the layer/substrate interface induced at HP–HT can be estimated [6] from the formula

$$\varepsilon_{HP-HT} = (a_S/a_{relax})\left[\Delta T(\alpha_S - \alpha_L) + \frac{1}{3}HP\left(\frac{1}{B_L} - \frac{1}{B_S}\right)\right]. \tag{1}$$

Here HP means the applied hydrostatic pressure, ΔT – the difference between the treatment temperature and the room one, a_{relax} is the relaxed lattice parameter of the layer material. The bottom indexes denote the layer (L) and substrate (S) materials.

The change in mismatch strain can be related to the misfit and threading dislocations density changes. The presence of the misfit and threading dislocations is revealed by the X-ray method in a different way. The misfit dislocations affect the X-ray diffuse scattering intensity and lateral lattice parameters of the layer material. The density of the misfit dislocations ρ can be calculated from the X-ray diffuse scattering intensity [7] and from the lateral lattice mismatch [8] via the formula

$$\varepsilon = \frac{(a_{\parallel} - a_{relax})}{a_{relax}} = \varepsilon_0 + b_{\parallel}\rho \tag{2}$$

where $a_{\parallel}$ means the lateral lattice parameter of the layer material and ε_0 is the strain for the pseudomorphic layer. Assuming the presence of 60° dislocations, the edge component of Burgers vector $b_{\parallel}$ is given by: $b_{\parallel} = \sqrt{2}\cos 60° a_{relax}/2$.

The threading dislocations cause broadening of the rocking curve and an increase in the rocking curve width at half maximum (FWHM).

2. Experimental

The epitaxial layers were grown by the molecular beam epitaxy (MBE) on the (001) oriented semi-insulating GaAs substrates.

The 1.5 μm thick homoepitaxial beryllium doped GaAs:Be layers were grown at the substrate temperature equal to 870 K. The heteroepitaxial InAs layers of about 5 μm thickness were grown at 850 K. The 1.5 μm thick AlGaAs layers of different Al content were grown at 950 K.

The samples were HP–HT treated for 1 h under hydrostatic argon pressure equal to 1.2 GPa. The HP–HT treatment temperature for GaAs:Be/GaAs, InAs/GaAs and AlGaAs/GaAs samples was 870 K, 670 K and 920 K, respectively.

The beryllium c_{Be} and hole N_p concentrations were measured by secondary ions mass spectrometry (SIMS) and the Van der Pauw method, respectively. The absolute values of Be concentration were calculated from the SIMS data (with an accuracy of about 20%) using the relative sensitivity factor.

X-ray investigations were carried out using a MRD-PHILIPS diffractometer in the double and triple configurations.

The strain state of the layers was determined from the lattice parameters by the Fewster method [9]. The lateral $a_{\parallel}$ and out-of-plane lattice parameters $a_{\perp}$ of the layer material were derived before and after the treatment from the symmetrical 004, 006 and asymmetrical 335 and 224 reflections. The relaxed lattice parameter a_{relax} was determined from the values of the out-of-plane and in-plane lattice parameters. Next, the misfit dislocation density ρ was calculated from the formula (2).

3. Results and discussion

3.1. GaAs:Be/GaAs homoepitaxial structure

Beryllium in GaAs:Be is incorporated substitutionally into the Ga sublattice (Be_{GA}), where it manifests an acceptor activity. The SIMS-detected Be concentration in the investigated samples was clearly higher than the concentration of surplus acceptors determined by the Van der Pauw method (Tab. 1). It means that the total Be concentration in the GaAs:Be layers was higher than that in the form of Be_{GA} incorporated into the Ga sublattice. It follows from the above that the surplus Be atoms occupy interstitial positions or create Be inclusions.

Table 1. Difference between the experimental and calculated values of the relaxed lattice parameters Δa for the as-grown and HP–HT trated GaAs:Be samples. (The beryllium c_{Be} and hole N_p concentrations were measured by SIMS and the Van der Pauw method, respectively).

c_{Be} [10^{19} cm^{-3}]	N_p [10^{19} cm^{-3}]	As-grown $a \pm 0.5 \times 10^{-4}$ [Å]	After HP–HT $\Delta a \pm 0.5 \times 10^{-4}$ [Å]
0.5	0.5	−0.5	−0.5
5	2.7	0.1	1.1
18	7.0	1.5	3.0
21.5	5.8	3.2	5.2

The decrease in the out-of-plane and relaxed lattice parameters of GaAs:Be is related to the different atom sizes of Be and Ga atoms (covalent radius of Be is equal to 1.11 Å, while that of Ga – to 1.22 Å) and to the hole concentration effect [10]. However, for the Be concentration higher than 0.5×10^{19} cm^{-3}, the experimentally determined decrease in the lattice constant value was smaller than that calculated, due to the presence of beryllium containing inclusions (calculations of the lattice constant were performed for the Be concentration obtained from electrical measurements). It is known that the presence of inclusions can result in an increase in lattice constant.

The layers were fully strained before and after the treatment. It means that the in-plane lattice parameters were the same as those of the substrate and remained unchanged also after the treatment. It means also that the misfit dislocations were not created during the HP–HT treatment. From among the out-of-plane and in-plane lattice parameters, the relaxed ones were calculated [11].

The increased relaxed lattice constant a_{relax} values (in comparison with those for the as-grown samples) were detected after the HP–HT treatment for the GaAs:Be samples with the Be concentration above 0.5×10^{19} cm^{-3}. The HP – HT treatment influences the defect structure of GaAs:Be. The HP – HT treatment, due to different compressibility and thermal expansion of the Be-containing precipitates (in relation to those of the matrix material), results in the creation of additional defects influencing the lattice parameter value. It is known that, at sufficiently severe HP – HT conditions, the stress at the precipitate/matrix interface can reach the critical value for emitting dislocations loops and other defects [12]. Indeed, the HP – HT treated samples indicate the increased lattice parameters (Tab. 1)

Our results suggest that a part of Be atoms creates, during the GaAs:Be structure growth, some Be-containing defects. Such assumption allows to explain the difference between experimental and calculated lattice parameters for the as-grown samples, as well as the increased lattice parameter after the HP – HT treatment.

3.2. InAs/GaAs and AlGaAs/GaAs heteroepitaxial structures

The bulk modulus of the heteroepitaxial InAs layer material (B_{InAs} = 62 GPa) and that of the GaAs substrate (B_{InAs} = 83 GPa), as well as their thermal expansion coefficients ($\alpha_{InAs} = 5.20 \times 10^{-6}$ K^{-1}; $\alpha_{GaAs} = 6.03 \times 10^{-6}$ K^{-1}), are distinctly different. So, one would expect the most pronounced effects induced by the HP – HT treatment just for the InAs/GaAs structure (see formula (1)).

The as-grown InAs layers were fully relaxed. Only the residual compressive thermal strain has been detected for the as-prepared InAs/GaAs structure as shown in Table 2.

Table 2. FWHM for 004 reflection and strain ε for the InGa/GaAs and AlGaAs/GaAs samples, as-grown and HP – HT treated.

Sample	FWHM [arcsec] for 004 reflection				Strain ε	
	As-grown		After HP – HT		As-grown	After HP – HT
	S	L	S	L		
InAs/GaAs	16	120	16	106	-3.7×10^{-4}	-1.13×10^{-3}
$Al_{0.4}Ga_{0.6}As/GaAs$	16	19	16	25	-5.7×10^{-4}	-6.9×10^{-4}
$Al_{0.7}Ga_{0.3}As/GaAs$	19	23	19	25	-1.05×10^{-3}	-1.10×10^{-3}

(S – substrate, L – layer).

The $2\theta/\omega$ scans of 004 reflection for the InAs layer before and after the HP – HT treatment are presented in Fig. 1. As a result of the HP – HT treatment, the out-of -plane lattice parameter increased from 6.06122 Å to 6.06655 Å. The lateral lattice parameter decreased simultaneously from 6.0565 Å to 6.0520 Å. The decreased value of the in-plane lattice parameter of InAs after the HP – HT treatment means that the layer became to be better matched to the substrate. The absolute value of strain increased after HP – HT treatment from 3.7×10^{-4} to 1.13×10^{-3} (Tab. 2). This effect is related to the decreased, by about 10^5 cm^{-2}, density of misfit dislocations.

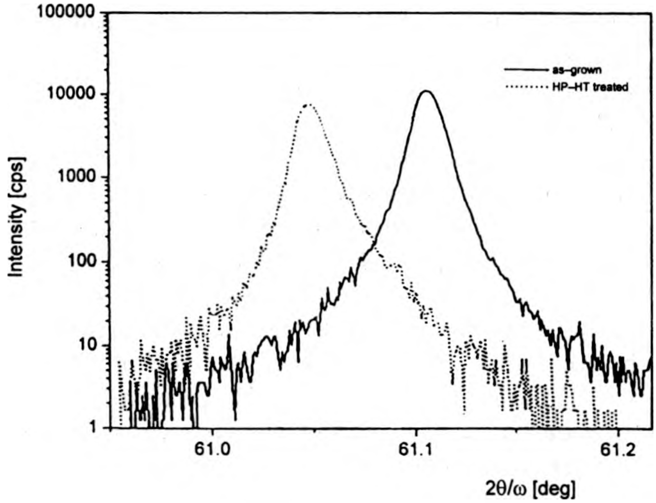

Fig. 1. The $2\theta/\omega$ scans of 004 reflection for the InAs layer; as-grown and after the HP – HT treatment.

It is necessary to admit that, for the InAs/GaAs system, the 29 – 40% decrease in FWHM (as dependent on the investigated sample) was found. Because no change of the curvature radius of the samples has been detected, the decreased FWHM value evidences the lowered density of threading dislocations.

During the HP – HT treatment the lattice mismatch in the InAs/GaAs samples decreased (from 7% at the layer growth temperature to 6%). It means that the InAs layer becomes to be better matches to the GaAs substrate. This structural change remained to be partially retained also at ambient conditions and therefore the

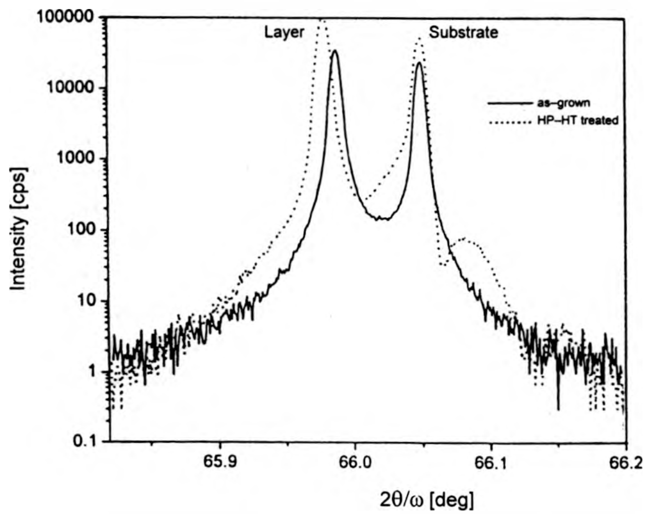

Fig. 2. The $2\theta/\omega$ scans od 004 reflection for the $Al_{0.4}Ga_{0.6}As/GaAs$ sample; as-grown and after the HP – HT treatment.

increased strain and decreased FWHM values were observed when the misfit and threading dislocation density decreased.

The heteroepitaxial AlGaAs layers were fully strained before the HT–HP treatment. The $2\theta/\omega$ scans of 004 reflection for the $Al_{0.4}Ga_{0.6}As/GaAs$ sample, before and after the treatment, are presented in Fig. 2. An increase in the out-of -plane lattice parameter by about 6×10^{-4} Å and a decrease in the lateral lattice parameter by about 3×10^{-4} Å for the $Al_{0.4}Ga_{0.6}As/GaAs$ and $Al_{0.7}Ga_{0.3}As/GaAs$ samples were found after the treatment. The effect of the treatment at 920 K – 1.2 GPa on the strain in the AlGaAs/GaAs structures of different compositions is presented in Tab. 2. The increased strain and the misfit dislocation density (the latter for about 10^4 cm^{-1}) were observed after the treatment. The rocking curve broadening of the layer in the HP–HT treated samples (Tab. 2) can be attributed to the presence of threading dislocations, as well as of other defects created during the HP–HT treatment (compare with above presented results for homoepitaxial GaAs:Be/GaAs).

The bulk modulus of AlGaAs is very close to that of GaAs ($B_{AlAs} = 83$ GPa, $B_{GaAs} = 81$ GPa) and therefore an effect of the HP–HT treatment on the defect structure changes in AlGaAs/GaAs has been much less pronounced than in the case of InAs/GaAs samples.

4. Conclusions

The changed defect structure of the samples caused by the HP–HT treatment was observed in the investigated structures after the release of pressure and temperature to ambient conditions.

For the GaAs:Be/GaAs samples no changes of the strain state and of the misfit dislocation density were detected. However, the presence of additional defects was observed after the HP–HT treatment. It means that the HP–HT treatment induced changes in the sample defect structure (newly created defects) can be considered as a kind of indicator of their primary defect structure. Using complementary electrical, SIMS and X-ray methods and applying the treatment at high hydrostatic pressure, it occurred to be possible to confirm the presence of Be-containing structural irregularities and to gain some insight into their nature.

The effect of HP–HT for the InAs/GaAs and AlGaAs/GaAs systems was dependent mostly on the strain generated during the treatment, due to the difference in thermal expansion and the bulk modulus of the layer and substrate materials. If the lattice mismatch at HP–HT (during the treatment) was smaller than that during the layer growth, the better match of the layer to the substrate was detected after the release of HP–HT to ambient conditions. In the case of InAs/GaAs system it resulted in the increased strain and in the decreased misfit and threading dislocations density. In the case of fully strained AlGaAs/GaAs samples, additional misfit and threading dislocations were created after the HP–HT treatment.

In summary, our work confirmed that the change in strain and in the misfit dislocation density can be expected only for the layered structures with different layer compressibility and thermal expansion in respect to that of the substrate. The HP–HT treatment affects also the defect structure of the layers and this effect depends on the nature and density of the primary existing defects.

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