ANALYZE OF DEFECTS IN InGaAs EPILAYER WITH HIGH-RESOLUTION X-RAY DIFFRACTION

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Molekulyar Şüa Epitaksiyası ilə alınan $In_{0.16}Ga_{0.84}As$:Be/ : GaAs hazırlanan epitaksial təbəqədə yaranan qüsurları yüksək həssasiyətli rentgen difraksiyası metodu ilə araşdlırıldı. Nümunədəki təbəqə içi və təbəqələr arası gərginliklər aşgar edildi. Bu qüsurlar cihazın işləmə keyfiyyətinə mühüm ölçüdə təsir edirlər. Nümunə üçün $In_{0.16}Ga_{0.84}As$ epitaksial təbəqə tədqiq edildi. Cünki, bu strukturda hərbir qüsur rahatca biri-birindən ayrılıqda ayrıntılı araşdırıla bilir. Təcrübi olaraq bu parametrləri görüntüləmək üçün dinamik nəzəri əsaslı hesablamalar edildi. ε_{II}, $\varepsilon_{\perp}$, ε_f , relaksasiya dərəcəsi R, İndimun miqdarı, epitaksial təbəqənin düzlem əyilmə bucaq qiymətləri və dislokassiyalar yuksək həssasiyətli rentgen difraksiyası ölçülərindən hesablandı.

Методом рентгеновской дифракции высокого разрешения (HRXRD) были изучены релаксация напряжения и последовательное накопление дефектов в эпислое $In_{0.16}Ga_{0.84}As$:Be, полученного на подложке GaAs методом молекулярно-лучевой эпитаксии,. Приведен анализ структуры дефекта, сильно влияющего на работу прибора. Для этого проведены исследования эпислоя InGaAs. Экспериментально для подтверждения полученных параметров проведены расчеты в рамках динамической теории для ε_{II}, $\varepsilon_{\perp}$, ε_f , R, с учетом содержания индия и дислокаций.

Be doped $In_{0.16}Ga_{0.84}As$ epilayer was grown on semi-insulating GaAs(100) substrate by molecular beam epitaxy. Strain relaxation and defects in the structure have been carried out using high resolution X-ray diffraction (HRXRD) technique has revealed the in-plane and out-of-plane strains in samples. The plane quality of the sample was investigated by analyzing of the full width at half maximum (FWHM) fluctuations depend on the increasing azimuth angle of the sample's reflections. Using the experimentally determined strain parameters, a dynamical theory based calculations have done. Parallel X-ray strain (ε_{II}), perpendicular X-ray strain($\varepsilon_{\perp}$), misfit(ε_f), degree of relaxation (R), x composition, tilt values and dislocations are calculated by HRXRD measurements.

I. INTRODUCTION

The lattice–mismatched GaAs based hetero-structures are of continual interest because of their application for high-speed electronic and optoelectronic devices. They offer also a basis for fabrication of a variety of low-dimensional and mesoscopic systems being a subject of current studies in solid-state physics. Several types of defects in these structures affect strongly the operating performance of the devices. Defects and strain relaxation in InGaAs/GaAs heterostructures have been studied by several researchers due to the effects on the electrical and optical properties of heterojunction devices [1-3]. Epitaxial growth of those heterostructures is accompanied by a strain in the epitaxial layer that results from a difference in lattice parameters between the substrate and the epilayer. If the thickness of the layer exceeds its critical value the strain is relieved by the formation of misfit dislocations. In heteroepitaxial semiconductor systems with zinc-blend structure and small lattice mismatch, grown on (001)-oriented substrates, orthogonal arrays of regular 60° misfit dislocations are formed at the interface. The misfit dislocations are accompanied by threading dislocations which propagate into the epitaxial layer. The presence of a network of dislocations often results in a characteristic undulating surface morphology known as cross hatch which occurs in many lattice-mismatched semiconductor systems [4-6].

High-resolution X-ray diffraction (HRXRD) is useful technique for the analyzing of defects for the semiconductor heterostructures and quantum dot (QD) structures [7]. For this type analysis, a high resoluted monochromotor and a movable axises in the reciprocal space are necessary for detections of the asymmetric plane reflections. Also, HRXRD technique is a general way to measure the composition of epitaxial semiconductor compounds [8-15].

In this study, using HRXRD system, we researched a defected InGaAs epilayer because of the each defect in an epilayer can be separated easily and investigated in the detailed. In the different symmetrical and asymmetrical planes, the defects were analyzed as depending of the azimuth angle.

II. EXPERIMENTAL DETAILS

p-type $In_{0.16}Ga_{0.84}As$ epilayer was fabricated on epi-ready semi-insulating (SI) GaAs (100) substrate in a V80H-MBE system using elemental sources for Ga, In and As beams. Beryllium was used as p-type doping source. As_2 beams are obtained by using cracker cell at 950°. The growth rate and reconstruction of the (100) surface were determined by RHEED oscillations. A transition of amorphous circular pattern to a 2x4 streaked pattern and the surface oxide desorption has been observed as the substrate is heated above 580°C. After then the substrate temperature was lowered to 560°C for growth of the entire epitaxial structure. During the epilayer growth, beam equivalent pressure (BEP) for In and Ga are kept as 2.7×10^{-5} mbar and 7.4×10^{-7} mbar, respectively. The V/III flow ratio was kept as 3. Growth rate of GaAs for buffer layer was 2.780Å/s. For the InGaAs epilayer, growth rate of GaAs and InAs were 1.80Å/s and 1.52Å/s, respectively. For the structure, 1000 nm GaAs buffer layer growth followed by deposition of a 995nm InGaAs layer.

The HRXRD measurements were performed by D8-Discover diffractometer equipped on the primary side with a

four crystalled Ge (220) monochromator for $CuK_{\alpha 1}$ X-ray beam (λ = 1.5406 Å) and a horizontal divergence slit with a width of 1mm. As regards the Si calibration sample, its best resolution was 16 arcsec. On the secondary side, the reflected light passes a horizontal slit with a width of 0.1 mm before entering the wide open scintillation detector. Rocking curves of the sample was measured by $\varpi / 2\theta$ scan (where ϖ and 2θ are the angles of the sample and detector relative to the incident X-ray beam) with a detector angular acceptance of $\sim 1^{\circ}$.

III. RESULTS AND DISCUSSION

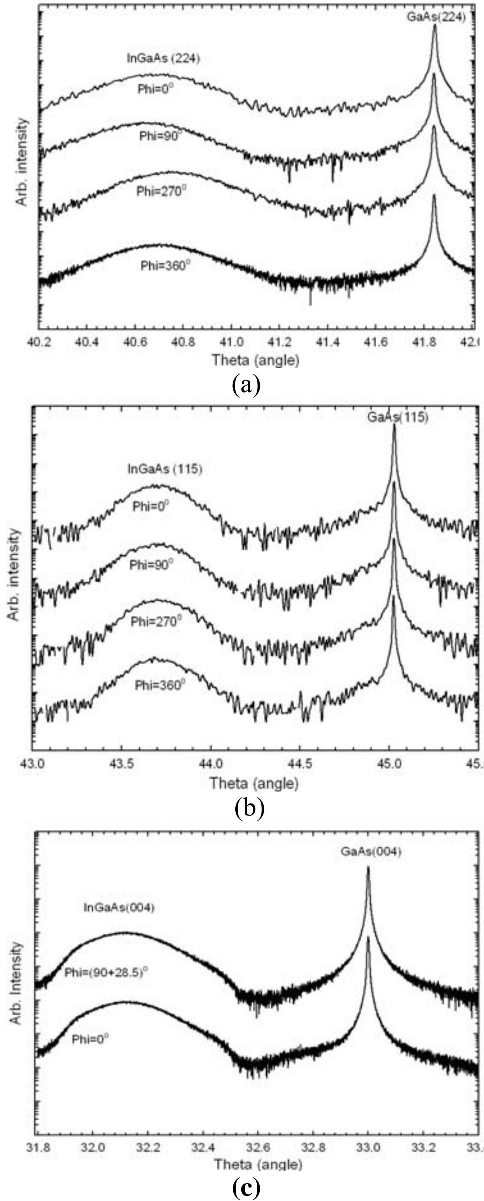

Fig.1. HR- XRD pattern (004), (115) and (224) reflections for the sample: (a) Layer to substrate peak separation for (224) reflection at four azimuths, (b) Layer to substrate peak separation for (115) reflection at four azimuths, (c) Layer to substrate peak separation for (004) reflection at two azimuths,

The $In_{0.16}Ga_{0.84}As$ epilayer was grown on GaAs(100) substrate be solid source molecular beam epitaxy. The structural parameters of the epilayer were determined using HRXRD. Profiles of all symmetric and several asymmetric scans were recorded in ω/2θ scans after optimizing z-height alignment, the tilt angles. Reflections (002)S, (004)S, (006)S, (115)A and (224) A (S = Symmetric and A = Asymmetric scan, respectively) were recorded for InGaAs epilayer sample. These reflections are scanned for the positions of all azimuth plane and they were given in Figs. 1a, 1b and 1c. As shown in these figures, two main peaks were observed. The peak with higher intensity comes from the GaAs substrate. Other peak originates from InGaAs epilayer. All scans are given to observe the minor changes, determining the structure of defect.

From the sizes and positions of the ω/2θ peaks, one can also deduce the information on crystal imperfections like point defects and dislocations and their interactions, as well as the changes of lattice parameters of the unit cell in an film, caused by misfit strain relaxation.

Azimuth angles and the FWHM of the layer peaks measured from the experimental (004), (115) and (224) ω-2θ scans were given in Table 1. FWHM values show a fluctuation for the different azimuth angles of different reflections of the sample. This fluctuation suggests that the plane quality changes with increasing azimuth angle of the sample's reflections.

The indium fraction (x) in the $In_xGa_{1-x}As$ epilayer determines the electronic and optical behaviors of devices, therefore accurate measurements of this parameter are of great importance. It is commonly calculated by

$$x = \frac{a_{InGaAs} - a_{GaAs}}{a_{InAs} - a_{GaAs}} . \quad (1)$$

where, a_i is lattice parameter of ith compound. Using eq. (1), the In composition of the sample was calculated as 0.164. The lattice parameters $a_{\perp}$ and a_{II}, which are perpendicular and parallel to the layer plane, respectively, were calculated from the Bragg law with reflection positions by [16,17]

$$2\sin\theta_B \cos\varphi = \frac{l\lambda}{a_{\perp}} \quad (2)$$

$$2\sin\theta_B \sin\varphi = \frac{\sqrt{h^2 + k^2}\,\lambda}{a_{II}} . \quad (3)$$

where θ_B is Bragg angle, φ is the angle between the diffraction plane and the sample surface, and λ is the wavelength of the X-rays.

Then, to find the x with different method, a mean lattice parameter of the obtained lattice parameters was used. In the different azimuth angle, all lattice parameters are given in the fifth and sixth columns of Table 1. The lattice parameters in the asymmetric planes show more changing than those of the symmetric planes, which explain the in-plane defects. The concentration can be also obtained as depending on elastic constant. The layer fractional mismatch with respect to the substrate, f, defined as $(a_{layer}-a_{sub})/a_{sub}$ is related through the elastic coefficients [3]

$$f = (\varepsilon_{\perp} + P\varepsilon_{II})/(1+P), \quad P = 2C_{12}/C_{11} \quad (4)$$

$$x = f a_{GaAs}/(a_{InAs} - a_{GaAs}) \quad (5)$$

From this method, the indium content value was found as 0.168 and the results of every two methods are well agreement.

Table 1.

For (004), (115) and (224) reflections FWHM, lattice constant and misfit values.

Reflection	Azimuth angle	FWHM (deg.) for GaAs	FWHM (deg.) for InGaAs	a(Å) for GaAs	a(Å) for InGaAs	Misfit
(004)	0°	0.00589	0.26900	5.65732	5.79297	0.02398
(004)	(90+28)°	0.00563	0.27300	5.65732	5.79345	0.02406
(115)	0°	0.25900	0.26000	5.65816	5.79324	0.02388
(115)	90°	0.01481	0.29900	5.65549	5.79018	0.02381
(115)	270°	0.29300	0.29400	5.65028	5.78417	0.02370
(115)	360°	0.00979	0.30300	5.65185	5.78691	0.02390
(224)	0°	0.00855	0.30800	5.65681	5.78722	0.02305
(224)	90°	0.01200	0.30600	5.66421	5.78828	0.02190
(224)	270°	0.00999	0.33900	5.63968	5.77621	0.02421
(224)	360°	0.01320	0.33700	5.64209	5.77388	0.02336

The data obtained using the ω/2θ reflections were analyzed for the out of plane and in-plane strains $\varepsilon_{\perp}$ and ε_{II}, respectively by least square analysis. Perpendicular (out-of-plane) $\varepsilon_{\perp}$ and parallel (in plane) ε_{II} strains are defined as follows, with respect to the substrate:

$$\varepsilon_{\perp} = \left(\frac{a_{\perp} - a_{sub}}{a_{sub}}\right) \qquad (6)$$

$$\varepsilon_{II} = \left(\frac{a_{II} - a_{sub}}{a_{sub}}\right) \qquad (7)$$

The $\varepsilon_{\perp}$ and ε_{II} strains are defined and their results are given in Table 2. The strains have large values and show the defects of epilayer.

With the four crystal (220) Ge conditioning the incident x-ray beam, the sample was scanned for (004), (115) and (224) reflections with 2° open detector in ω-2θ mode at four equivalent azimuthal settings.

Degree of relaxation R are calculated with help of the parallel X-ray strain and misfit (ε_f) by

$$R = (a_{II} - a_s)/(a_f - a_s) = (\varepsilon_{II}/\varepsilon_f)100\% \qquad (8)$$

$$\varepsilon_f = (a_f - a_s)/a_s \qquad (9)$$

a_f, bulk equivalent or unstrained lattice constant and a_s is the lattice constant of substrate, is calculated from the x- ray strains using the linear elasticity theory [2]. The obtained relaxation degree equals approximately to 1%. As depending on the azimuth angle, the calculated misfit values are given in the last column of Table 1. They change well after second digit of the misfit value in the different azimuth angles.

From layer to substrate peak separation at these settings, the layer to substrate tilt angle, $T_{L,S}$ is estimated from

$$T_{L,S} = [(\Delta_1 - \Delta_3)^2 + (\Delta_2 - \Delta_4)^2]/4 \qquad (10)$$

where Δ is the peak separation for four azimuthal settings[18-20]. So the tilt between the epitaxial layers and the substrate may change the angular separation between the diffraction peaks. To eliminate the effect of this tilt, three sets of (004), (115) and (224) scans were performed at two orientations by rotating the sample by 180° around the [001] axis. The average angular separation of the peaks for each set of scans was calculated.

Tilt angels were calculated along (004), (115) and (224) planes and were less than 0.1°, and these values are general result [21]. Tilt of the (004) direction is the lowest one, 9.000 x10^{-6} due to the asymmetric planes. Its reason is that the asymmetric planes are affected very much from strain.

Average spacing D of 60° misfit dislocation was calculated from the measured in-plane mismatch using

$$D = a_s / \sqrt{2}\varepsilon_{II}\,. \qquad (11)$$

Table 2.

Strain parameters, In composition, tilt angle and relaxation degree of the InGaAs/GaAs epilayer.

In content	Mean strains		Tilt angle for			Relax. degree	Average spacing of D	Dis. Density
x	ε_{II}	$\varepsilon_{\perp}$	(004)	(115)	(224)	R(%)	D(Å)	ρ
0.164(eq.1)/ 0.168(eq5)	0.02235	0.02388	9.000x10^{-6}	7.200x10^{-5}	9.928x10^{-3}	~1	358	2.79x10^{+5}

Analyses of the HR-XRD measurements of the above sample in the symmetric and asymmetric reflections are determined. Average spacing D of 60° misfit dislocation was calculated as 358 Å. When this value is compared with distance inter two atoms in the lattice, it is very large. Also inverse of it determines the dislocation density ρ=*1/D*,

$2.79x10^{+5}cm^{-1}$. This large value results in the cross hatch. Also for symmetric and asymmetric planes, D and ρ are calculated as depending on azimuth angles. Their values show a fluctuation with increasing azimuth angles in third and forth column of the Table 3. The sample details and calculated parameters were collected in the Table 1and 2.

We can estimate the sample curvature and linear dislocation densities with the data we have

$$f = \varepsilon_{\perp} - [2\nu/(1-\nu)](1+6\frac{t}{t_0})(\frac{t_0^{\ 2}}{6t_l R}) \tag{12}$$

$$(N)b_l = \varepsilon_{\perp} - [(1+\nu)/(1-\nu)](1+\frac{6t_l}{t_0})(\frac{t_0}{6t_l R}), \tag{13}$$

where t_0 and t_l are the substrate and layer thickness, R the radius of curvature of the layer, N the linear dislocation density, b_l the component of the Burgers vector along the layer interface, and ν the Poisson ratio. b_l is approximately $a_{layer}/2$.

Table 3.

For (004), (115) and (224) reflections averaging spacing of misfit dislocation and dislocation density values.

Reflections	Azimuth angle	Averaging spacing of misfit dislocation	Dislocation Density(cm^{-1})	
		D(cm)	1/D (cm^{-1})	ρ(cm^{-1})(eq. 13)
(004)	0º	$1.668x10^{-6}$	$5.990\ x10^{+5}$	
(004)	(90+28)º	$1.663\ x10^{-6}$	$6.010\ x10^{+5}$	
(115)	0º	$1.676\ x10^{-6}$	$5.970\ x10^{+5}$	$0.594\ x10^{+5}$
(115)	90º	$1.679\ x10^{-6}$	$5.960\ x10^{+5}$	
(115)	270º	$1.686\ x10^{-6}$	$5.930\ x10^{+5}$	
(115)	360º	$1.672\ x10^{-6}$	$5.980\ x10^{+5}$	
(224)	0º	$1.735\ x10^{-6}$	$5.760\ x10^{+5}$	$0.110x10^{+5}$
(224)	90º	$1.829\ x10^{-6}$	$5.470\ x10^{+5}$	
(224)	270º	$1.647\ x10^{-6}$	$6.070\ x10^{+5}$	
(224)	360º	$1.708\ x10^{-6}$	$5.850\ x10^{+5}$	

Linear dislocation densities were calculated from eq.12 and eq.13 with help of the calculated structural parameters for (115) and (224) reflections and their values were given in last coulomb of Table 3. When the found dislocation values are compared with ρ=1/D in the same table, they are smaller in the same order. In addition, the curvature radius is found from eqs 12 and 13 as 3.8 meter for (115) reflection. Also, curvature radius are obtained proportionally for (224) reflections and found as 0.7 meter. Distinctly when we consider the approximately empirical relationship, curvature radius was calculated by [21]

$$\omega_{FWHM} = 52\frac{L}{R} \tag{14}$$

where L is the illuminated length, R is radius of curvature, and W_{FWHM} is full width at half maximum of the (006) reflections in radian unit. Curvature value from this equation was found 5.8 m, which is higher value than those obtained from symmetric reflections.

An in-plane Φ-scan was also taken by rotating the sample around its surface or selected asymmetric plane -normal direction to investigate the in-plane alignment of the GaAs film. Here, we scanned for the (004), (115) and (224) and their reflections are given in the Fig. 2. The reflections of these planes are repeated at 90º except the (004) plane because of azimuth planes are not approximately perpendicular. They confirm the cubic structure of the InGaAs epilayer. Also we can get information about defects of the azimuth angles. As shown in figure 2, the intensities of the reflections have about the same amplitude, which shows that the azimuth planes are perpendicular to the reflections plane.

IV. CONCLUSIONS

InGaAs/GaAs epilayer structure was grown on a GaAs substrate by solid source molecular beam epitaxy (MBE). A small slipping on the peaks for ω-2θ measurements was determined by investigating the selected reflections for the epilayer. This slipping causes defects like tilt, strains and dislocation. The detailed defect analysis is done and these defects are certainly changed the azimuth angles of the symmetric and asymmetric planes. Higher dislocation and mismatch values are weakly fluctuated in same order. High resolution X-ray diffraction (HRXRD) measurements showed that the fluctuation of tilting angles of local crystal planes in the epilayer. This situation is because of the difference in relaxation of lattice-mismatch between InGaAs and GaAs layers for the sample.

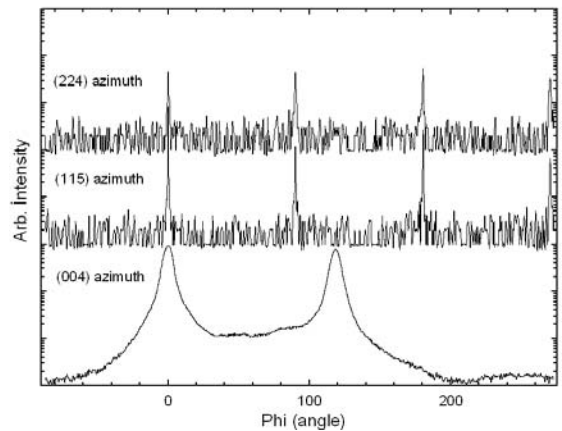

Fig. 2. Azimuths for (004), (115) and (224) reflections

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