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Investigation by synchrotron radiation X-ray topography of lattice tilt formation in partially released InGaAs/GaAs compositionally graded layers

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Abstract

The synchrotron radiation plane wave topography and the Rutherford backscattering technique have been used to investigate the lattice tilts between substrates and layers introduced by the preferential orientation of the Burgers vectors of the misfit dislocations in compositionally graded InGaAs/GaAs heterostructures. A monotonic change of the lattice tilt along the sample surface producing in average a concave curvature of the buffer layer lattice has been found with a nearly complete alignment of the Burgers vector of the misfit dislocations at the sample edges. The topographic observations showed that the buffer layers can follow a nearly continuous curvature or can be sub-divided in large domains with different average lattice tilt. The models recently proposed for the formation of tilt in partially released structures are not able to explain the present observation of a lattice tilt varying coherently along the sample surface. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

In recent years there has been a considerable interest in studying compositionally graded buffer layers suitable for the preparation of devices based on mismatched semiconductor heterostructures (see for instance Refs. [1,2], and references therein). In particular, compositionally graded buffer layers have been demonstrated to be particularly efficient in reducing: (i) the misfit dislocation density near the top of the buffer and in the active part of the heterostructure, and (ii) the threading dislocation density, which affects negatively the device

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performance and reliability (see for instance Ref. [3], and references included).

According to the model first proposed by Mazzer et al. [4], the formation of arrays of parallel misfit dislocations at the interfaces in partially relaxed heterostructures can lead to significant misalignments (tilts) between the substrate and the layer lattices if there is an unbalance of the misfit dislocation Burgers vector components perpendicular to the interface. Such a result can also be deduced from the more general Frank rule (see for instance Ref. [5]) which establishes the lattice tilt across a grain boundary for a given network of dislocations in the boundary.

Although there is no experimental evidence of detrimental effects on the quality of the buffer structure, lattice tilt is an interesting phenomenon because it is closely connected with the mechanism of misfit dislocation formation which has not been completely understood to date. Lattice tilts have been evidenced in partially relaxed heterostructures independent of the growth mode and lattice mismatch values (see for instance, Refs. [6–8]). In particular, compositionally step graded buffers seem to exhibit much larger values of the lattice tilts [2,9–11], as compared to single layers having the same average composition.

In the present work for the first time the synchrotron radiation topography has been used to investigate lattice tilt variations in compositionally graded InGaAs/GaAs buffer layers grown on nominally (0 0 1) oriented GaAs substrates. The results of such an investigation are discussed on the basis of recent models proposed for the formation of lattice tilts in partially released single heterostructures.

2. Experimental procedure

InGaAs buffers with different composition profiles (see Table 1) were grown on semi-insulating GaAs wafers (0 0 1) oriented within 0.1° by solid source molecular beam epitaxy (MBE) at 500°C using As_4 beams. Composition gradients were obtained by varying the In and the Ga fluxes using a computer-controlled procedure that sets the cell temperatures. Prior to each run, the In and Ga fluxes were calibrated by RHEED intensity oscillations from InAs and GaAs layers on InAs and GaAs substrates, respectively. The growing-surface temperature was maintained at 500°C during the growth. The samples have dimensions of $13 \times 16\ \mathrm{mm}^2$ and are kept in rotation during the growth to avoid composition disuniformities. The As_4 beam equivalent pressure (BEP) was 9.5×10^{-6} Torr, while the As_4/Ga BEP ratio was 18. Other details of the growth procedure are reported by Bosacchi et al. [12].

Table 1
Summary of the composition profiles of the $In_xGa_{1-x}As$/GaAs compositionally graded buffers investigated by X-ray topography

Sample	Composition gradient	$X_{\min}$(In)	$X_{\max}$(In)	Thickness (μm)
#977	Six constant steps	0.0	0.30	2.36
#914	Square root	0.005	0.350	2.33
#916	Linear	0.10	0.349	1.66

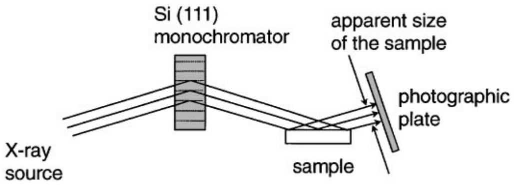

Fig. 1. Scheme of the diffraction and topography experiments at the ESRF synchrotron. Due to the low value of the Bragg angle the apparent size of the sample in the topograph is reduced by a factor of 0.19 in the direction parallel to the scattering plane.

Topography and high-resolution X-ray diffraction experiments were performed at ID19 beamline at ESRF of Grenoble (France). The samples analysed by X-ray diffraction and topography had typical dimensions of $6 \times 16\ \mathrm{mm}^2$ and were cut from the original pieces.

X-ray diffraction profile measurements and topography of both the buffer layers and the GaAs substrates were taken in the 0 0 4 symmetrical reflection geometry corresponding to a Bragg angle of 11.5° (Fig. 1) for the 0.53 Å wavelength selected by a Si 1 1 1 monochromator set in the transmission diffraction geometry.

A direct image of the incident beam was taken in order to distinguish instrumental effects (for instance, due to the phase contrast introduced by the beryllium window) from sample features. Both in the topography and in the diffraction profile measurements the X-ray beam section was larger than the sample size.

The dislocation distribution in the undoped GaAs substrate has been investigated by taking white beam Laue X-ray topographs in the transmission mode. The samples were mounted on the goniometer head with the [0 0 1] surface normal nearly parallel to the incident beam. In this geometry the beams diffracted at low Bragg angles correspond to short wavelengths and to reflecting planes nearly perpendicular to the sample surface.

Rutherford backscattering spectrometry (RBS) in channelling mode at the 2MV VdG accelerator for the AN2000 at LNL Lab (Italy) has been used to determine the local lattice tilt of the [0 0 1] axis. Assuming the centre of the samples as a reference, the maps of the tilt have been obtained by repeating the measurement in several points of the sample.

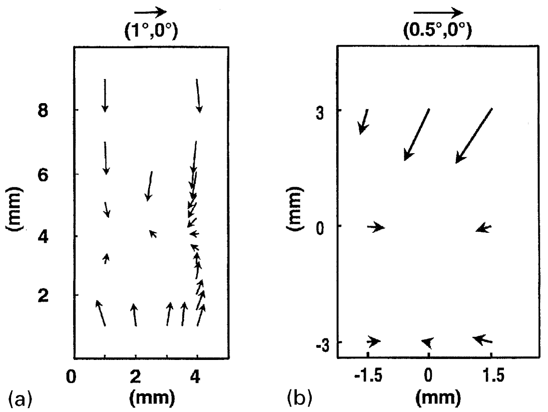

Fig. 2. (a) and (b) maps of the local angular deviation from the [0 0 1] lattice direction determined by ion channelling RBS in different position of samples #977 and #916 (see Table 1). The arrows give the magnitude and the projection of the lattice tilt on the (0 0 1) surface plane. The vertical direction correspond to the [1 1 0] crystallographic direction.

3. Results and discussion

3.1. RBS analysis in the channelling mode

Figs. 2a and b shows the maps of the tilt measured in the step-graded composition sample #977 and linear composition-graded sample #916 of Table 1. The arrows represent the projection of the [0 0 1] axis on the surface plane. Since the substrate curvature was found to be lower than 0.1°/cm as estimated by X-ray topographs in reflection mode and RBS measurements taken at the back surface of the sample, the deviations of the [0 0 1] crystallographic direction reported in Figs. 2a and b describe real changes of the buffer layer tilt with respect to the substrate. It is worth to note that this tilt changes monotonically over the sample area and reaches the maximum values at the [1 1 0] sample edges. Such monotonic change of lattice tilt corresponds to a concave curvature of the buffer layer lattice. For instance, the sample of Fig. 2a exhibits a curvature of 2.5°/cm along the [1 1 0] direction.

The samples of the present work belong to a larger set of InGaAs/GaAs composition-graded

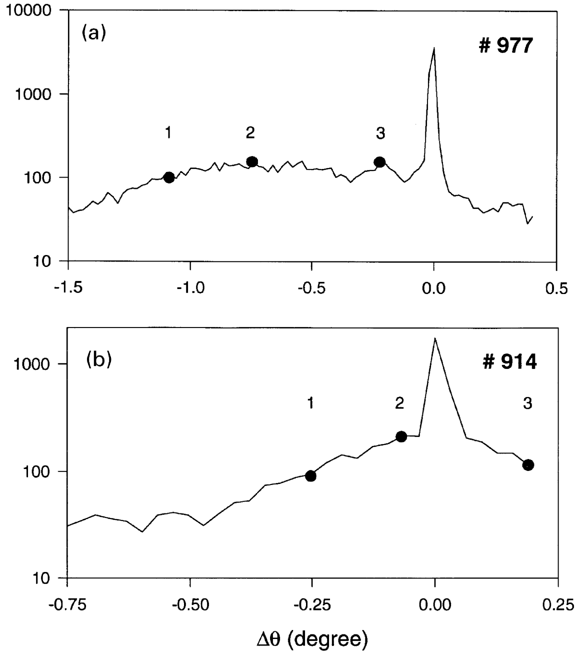

Fig. 3. X-ray diffraction rocking curves taken in the 0 0 4 symmetrical geometry near the GaAs Bragg angle of 10.9°. (a) and (b) refer to samples #977 and #914, respectively.

samples, in which Romanato et al. [13] systematically detected such monotonic lattice tilt change.

Although in InGaAs/GaAs wafers lattice tilts changing randomly with the sample position have been already observed [14], only composition-graded samples seem to exhibit such a regular curvature of the layer lattice.

3.2. X-ray diffraction and topography observation

Fig. 3 reports typical X-ray rocking curves of composition-graded InGaAs/GaAs samples described in Table 1. As visible in the curves of Fig. 3, the width and position of the buffer layer peak is greatly influenced by the change of lattice tilt along the sample surface so that a simple interpretation of

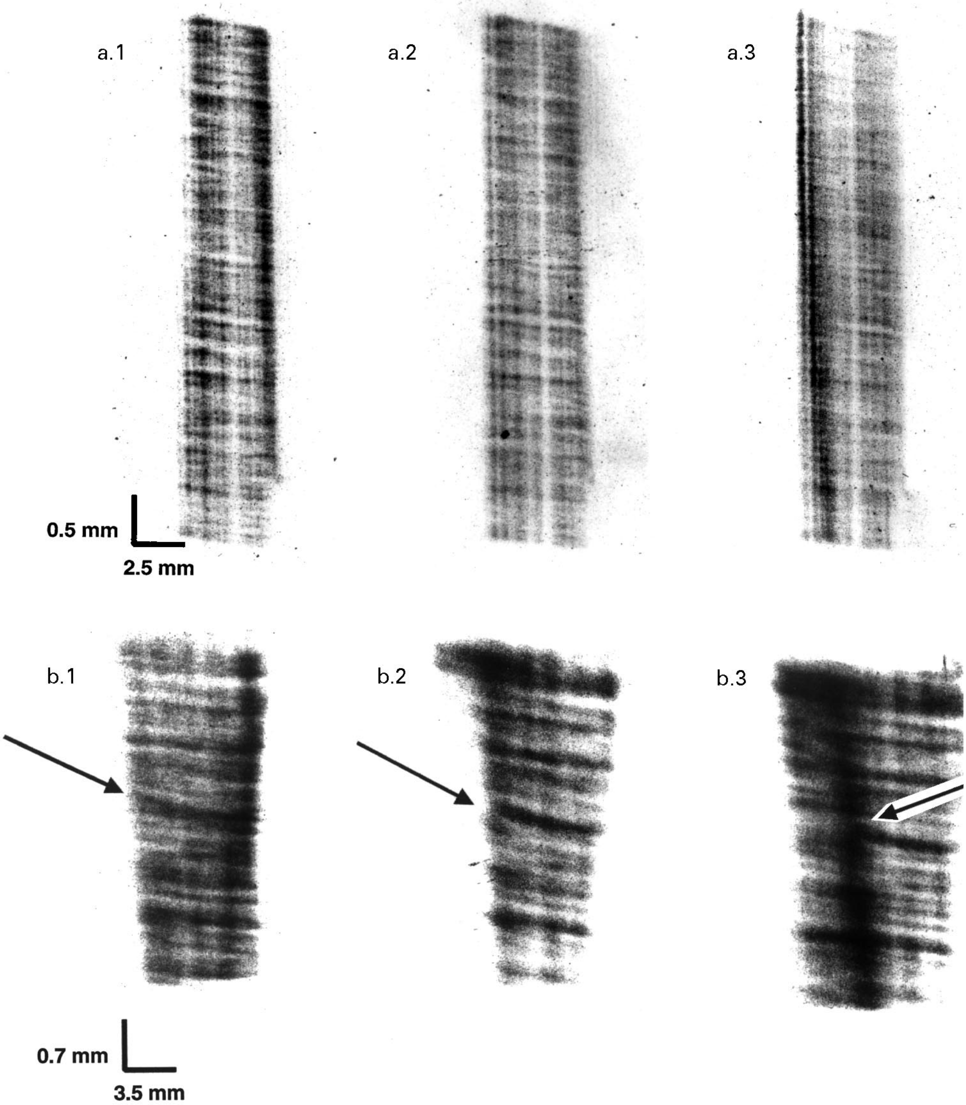

Fig. 4. Plane wave topographs of samples #977 and #914. The exposures a.1, a.2 and a.3 are taken at points 1, 2 and 3 of rocking curve 3a (Fig. 3a), whereas b.1, b.2 and b.3 correspond to points 1, 2 and 3 of rocking curve 3b (Fig. 3b). The arrow in Fig. 4b indicates the boundary of a large tilt domain.

the peak broadening can lead to a wrong estimation of In content. This is by far evident in Fig. 3c, where the high angle tail of the buffer layer peak lies on the left of the substrate peak, where diffraction from the InGaAs buffer layer is not expected due to the larger InGaAs lattice parameter with respect to GaAs.

Fig. 4 reports a double series of exposures taken at different angular positions of the rocking curves of Fig. 3.

3.3. Interpretation of contrast of X-ray topographs

The change of contrast among exposures recorded at different angles of incidence reveals parts of the buffer layer diffracting at different angles of incidence. In particular, the shift of the maximum of the diffracted intensity from the left to the right side of the topograph of Fig. 4a when decreasing the angle of incidence corresponds approximately to an average 2°/cm concave curvature of the buffer layer, in satisfactory agreement with the value of 2.5° obtained from channelling experiments.

All the topographs evidence a diffused cross-hatch contrast along the [1 1 0] and [1 $\bar{1}$ 0] directions, partially influenced by the phase contrast induced by the Be window of the beamline. Even if in the present case, the dislocation spacing lower than the topography resolution should produce no visible contrast, Argunova et al. [15] and Fewster et al. [16] have shown that in partially released heterostructures the topographic contrast is due to the local fluctuation of misfit dislocation (MD) density. Therefore, the lattice tilts induced by the local changes of dislocation densities rather than the threading dislocations [17] are mainly responsible for the peak broadening.

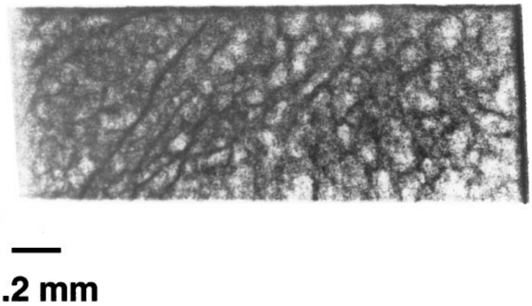

Fig. 5. White beam topograph of sample #914 in the transmission mode corresponding to diffracting planes nearly perpendicular to the (0 0 1) surface of the sample. The dark lines forming cellular structures correspond to the typical arrangement of dislocations in undoped GaAs Czochralski wafers.

The second group of topographs of Fig. 4 evidence the formation in the buffer layers of macroscopic grains having different average lattice tilt. The wall of one of these large domains nearly parallel to a (1 1 0) edge of the sample is clearly visible in Fig. 4b. The comparison with the Laue topograph of the substrate in Fig. 5 does not evidence any correlation between the two images, but only dislocations forming the typical cellular structure. A few MD bundles which stop at the wall of a domain are visible in detail in Fig. 4b. This indicates a possible correlation of the average MD line length with the domain size, which is of the order of several mm in Fig. 4b. It is worth noting that the formation of domains with different tilt produces on an average a concave curvature of the lattice.

3.4. Models for the tilt formation in partially relaxed heterostructures

The observed lattice curvature of the buffer can be described in terms of the distribution of the Burgers vectors of the MD network. According to Mazzer et al. [4], tilts between substrates and epilayers arise from the unbalance between the dislocation densities ρ^+ and ρ^- of MDs with opposite component of the Burgers vector perpendicular to the interface. The lattice tilt α is given by

$$\alpha = (\rho^+ - \rho^-)\cdot b^\perp = \Delta\rho \cdot b^\perp \qquad (1)$$

in which $b^\perp = 2.82$ Å for perfect $a/2[1\,1\,0]$ dislocations in a GaAs crystal. For a change of tilt of 2°/cm, the difference in the quantity $\Delta\rho$ for the dislocation densities at points A and B at a distance of 1 cm from each other is $\Delta\rho_B - \Delta\rho_A = 1.24 \times 10^6\ \text{cm}^{-1}$.

The comparison of such dislocation density with the MD density deduced from RBS measurements of the residual strain shows that at the [1 1 0] sample edges almost all the MDs have the same

Burgers vector component perpendicular to the interface [13]. For instance, in sample #916 where a residual strain at the top layer $\varepsilon = 6.3 \times 10^{-3}$ has been determined [18], the amount of misfit $f - \varepsilon$ accommodated by the buffer layer is given by

$$f - \varepsilon = 1.88 \times 10^{-2} \tag{2}$$

corresponding to a density of 60° $a/2\langle 1\,1\,0\rangle$ dislocations $\rho = 9.41 \times 10^{5}\ \mathrm{cm}^{-1}$, distributed over the buffer layer thickness and comparable to $\Delta\rho_{\mathrm{B}} - \Delta\rho_{\mathrm{A}}$.

To date several mechanisms for the formation of MDs with preferential Burger vector orientation have been proposed.

According to the model of Ayers et al. [11], lattice tilts arise from the preferential generation of MDs with larger Burgers vector component in the interface plane, which occurs at interfaces inclined with respect to the nominal (0 0 1) plane. However, the existence of tilt which is not correlated to the off-cut direction or layers grown on nominally oriented substrates has also been reported [14,19]. In the present case a lattice tilt depending on the position excludes such mechanism since for a given sample, the small unintentional off-cut angle was almost constant over the sample surface.

Kang et al. [19] observe that the elastic energy associated to the strain field of the MD network is reduced if the Burgers are preferentially oriented. According to this model lattice tilts should spontaneously occur over large areas of the wafer to reduce the elastic energy of the system. This again cannot account for the nearly continuous curvature found in many samples.

Riesz [20] proposed that the tilt may arise from the distribution of the Burgers vectors of the substrate threading dislocations. Furthermore, in an X-ray topography study of an InGaAs/GaAs single heterostructure, Barnett et al. [21] found a distribution of MDs which could be explained by a preferential orientation of the Burgers vectors of threading dislocations in the substrate. In fact, a preferential orientation of the Burgers vector component perpendicular to the (0 0 1) surface of the wafer has also been observed in Czochralski-grown GaAs crystal with etch pit density in the range 10^{4}–$10^{5}\ \mathrm{cm}^{-2}$ [22].

Nevertheless, no obvious correspondence is found between the structure of the tilt domains in the buffer and the cell structure of the substrate as seen in the comparison of topographs of Fig. 4b and the Laue topograph of Fig. 5, leading to the conclusion that the majority of the MDs develop independently on the initial distribution of the TDs.

4. Conclusions

The synchrotron radiation topography at ESRF facility in Grenoble has been used to study compositionally graded InGaAs/GaAs buffer layers. The analysis of the topographic images evidences that the distribution of MDs in the structures is not uniform, on a microscopic scale, no matter on the type of composition grading. The topographs contrast showed thin bands determined by bundles of MDs lines; in some samples very large independent grains were evidenced.

Large variations of lattice tilts, coherent over the whole sample dimensions ($6 \times 16\ \mathrm{mm}^{2}$) have been detected by RBS and X-ray topography in differently compositionally graded buffer layers grown on nonintentionally miscut substrates. The extent of such tilt variation shows that the normal components of the MD Burgers vector can be completely reversed over distances of few centimeters and are almost completely aligned in opposite directions at the sample edges.

Such observation permits to exclude that lattice tilts in the sample investigated is given by the surface off-cut, by a spontaneous alignment of Burgers vector or by the initial distribution of Burgers vector in the substrates, as proposed in the literature.

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