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# Characterization of $Al_{0.047}Ga_{0.953}Sb$ layers grown on GaSb using reciprocal space maps

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ABSTRACT

Structures of $Al_{0.047}Ga_{0.953}Sb$ layers on GaSb (100) substrates were studied by high resolution X-ray diffraction (HRXRD) using reciprocal space maps (RSM) and rocking curves around the (004) and (115) reflections. The layers were grown at 450 °C with a supersaturation of 10 °C in a conventional Liquid Phase Epitaxy (LPE) system varying the growth time from 1 to 4 min resulting in an increment of thickness. It was found that tilt, relaxation and dislocation density of the layers can be calculated using its rocking curves and reciprocal space mapping and it is found that these characteristics are influenced by the thickness of layer.



## 1. Introduction

The $Al_xGa_{1-x}Sb$ alloy is an attractive material because of its optoelectronic properties with potential applications in devices operating in the infrared region, such as photodetectors, lasers and light emitters [1–3]. The direct bandgap of this alloy can be tuned between 0.72 and 0.93 eV by changing its composition from $x=0$ to $x=0.4$. With $x=0.047$ the alloy can operate around the wavelength of less attenuation in optical fibers at 1.55 μm [4]. However, in order to fabricate efficient optoelectronic devices is essential to have a material with high crystalline quality with a low point defects concentration and that absorbs or emits light efficiently [5]. Unfortunately, in many cases the AlGaSb layers grown on GaSb are strained owing to their mismatch, showing structural defects.

By controlling the growth conditions one may grow epitaxially a pseudomorphic AlGaSb layer on a GaSb substrate until the thickness of the layer reaches a critical value, $h_c$, which depends upon both the aluminum content, $x$, and the growth temperature, $T_C$ [6,7]. If this critical thickness is exceeded, it becomes energetically favorable for the stress in the epilayer to be relieved through the formation of 60° a/2 misfit dislocations at the AlGaSb/GaSb interface.

Reciprocal space mapping is often used to investigate the structural properties of epitaxial thin films (layer tilt, lattice relaxation, composition and quality of structures) [8], and the transition from the fully strained to the relaxed state can also be investigated with the help of reciprocal space maps (RSMs) [9,10]. However, to our knowledge this technique has not been applied to the characterization of AlGaSb layers on GaSb.

Based on the theory developed by Kaganer et al. [11] and geometrical calculations of Chauveau et al. [12] it is possible to find, quantitatively parameters such as layer tilt, from high resolution RSMs. In this paper we analyze by means of HRXRD, the microstructure of partially relaxed $Al_{0.047}Ga_{0.953}Sb$ epilayers on GaSb (100) substrates. Four epitaxial samples of the same composition and different thickness were investigated to analyze the structural parameters such as the relaxation, tilt and composition.

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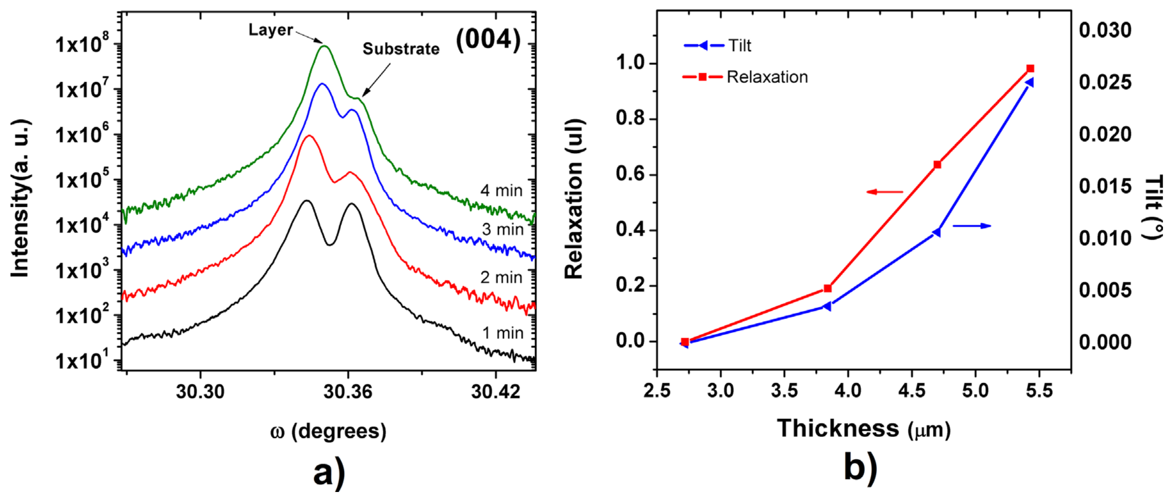


**Fig. 2.** (a) Rocking curves around the 004 Bragg reflection of the $Al_{0.047}Ga_{0.953}Sb$ epilayers on GaSb. The growth time of the layers varies from 1 to 4 min, (b) Estimated values of relaxation and tilt from the assymmetric 115 rocking curves and 004 RSMs for $Al_{0.047}Ga_{0.953}$/GaSb layers in function of thickness.

## 2. Experimental methods

$Al_{0.047}Ga_{0.953}Sb$ epilayers were grown on GaSb (100) substrates in a conventional Liquid phase epitaxy (LPE) system with a horizontal quartz furnace at 450 °C. The growth solution was a Ga-rich liquid mixed with GaSb and Al. To obtain an alloy with 4.7% of Al, the composition of the liquid solution was determined using the phase diagram and thermodynamic parameters for AlGaSb published by Elyukhin et. al. [13]. The solution and substrate are brought in contact after supercooling the solution by $\Delta T = 10$ °C, the contact times were 1, 2, 3 and 4 min.

Measurements of layer thickness were made using a scanning electron microscope (SEM) JEOL JSM-6610LV, using maximum electron energy of 20 keV. High-resolution X-ray diffraction measurements on the $Al_{0.047}Ga_{0.953}Sb$ epilayers were performed using a Bruker D8 Discover diffractometer at room temperature. The incident CuKα1 radiation ($\lambda$(CuKα1) = 1.54056 Å) was employed in combination with a V-groove Ge monochromator. Symmetric and asymmetric rocking curves for the (004) and (115) reflections were obtained.

Reciprocal space maps (RSMs) around the symmetrical (004) of the GaSb substrate were recorded with a scintillation counter detector to achieve sufficient resolution for the measurement.

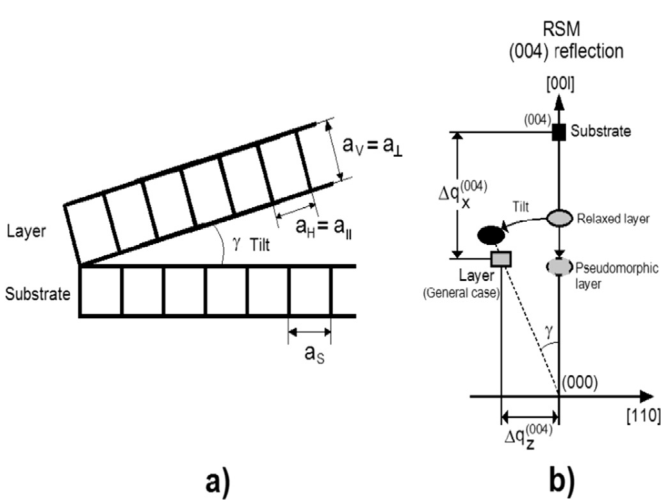


**Fig. 1.** (a) Schematic of a partially relaxed epilayer on a substrate tilted by $\gamma$ . In-plane and out-of-plane lattice constants are denoted $a_{\parallel}$ and $a_{\perp}$, respectively. (b) Schematic of reciprocal space for (004) RSM. Epilayer tilt causes rotation by $\gamma$ with respect to the origin. The epilayer coordinates relative to the substrate coordinates are defined as $\Delta q_x^{(004)}$ and $\Delta q_z^{(004)}$.

## 3. Theory

Reciprocal space maps contain a wealth of information on the structure of the layer and the substrate. Some information can be extracted by means of simple operations with the RSM, such as the peak position analysis. Misorientation or tilt of epitaxial layers relative to the substrate has been observed in lattice-mismatched epitaxy by others researchers [14,15]. These changes in orientation can be a result of two different causes. The first one is the miscut of the substrate which introduces surface steps. The second cause of tilting is the introduction of an array of dislocations during relaxation [16].

The epilayer coordinates in the reciprocal space strongly depend on their strain state and on the tilt of their crystallographic planes with respect to the substrate. Fig. 1(a) shows a schematic of a partially relaxed layer on a substrate with layer tilt $\gamma$. Fig. 1(b) is a schematic of substrate and layer positions in reciprocal space.

In general, the displacement of epilayer's coordinates in the reciprocal space has two components: a translation, due to the change from a untilted fully relaxed state to a compressively strained one. And a rotation angle, $\gamma$, around the origin due to the epilayer's crystallographic $\gamma$. The substrate material is assumed to be unstrained. Therefore, the substrate coordinates serve as reference position in the reciprocal space. The coordinates of the epilayer ($L$) in (004) RSM, expressed relative to the substrate coordinates ($S$) by $\Delta q_i^{(004)} = \Delta q_{i,L}^{(004)} - \Delta q_{i,S}^{(004)}$ with $i = x, z$ (004) RSMs allow the extraction of the tilt $\gamma$ between epilayer and substrate lattice planes. For each layer it is derived separately by the following geometrical relation [12] with the substrate lattice constant $a_S$:

**Table 1**
The values of Al content, $x$, relaxation degree, FWHM (004), cell parameters, $a_{\perp}$ and $a_{\parallel}$ and layer thickness, $d$, for different layers.

| Growth time (min) | $x$ (%) | $a_{\parallel}$ (Å) | $a_{\perp}$(Å) | Relaxation (%) | FWHM (arcsec) | $d$ (μm) |
|---|---|---|---|---|---|---|
| 1 | 4.58 | 6.09927 | 6.09591 | 0.4 | 25 | 2.72 |
| 2 | 4.68 | 6.09904 | 6.09625 | 19.2 | 29 | 3.84 |
| 3 | 4.75 | 6.09858 | 6.09711 | 61.3 | 43 | 4.70 |
| 4 | 4.62 | 6.09714 | 6.09710 | 98.3 | 68 | 5.43 |

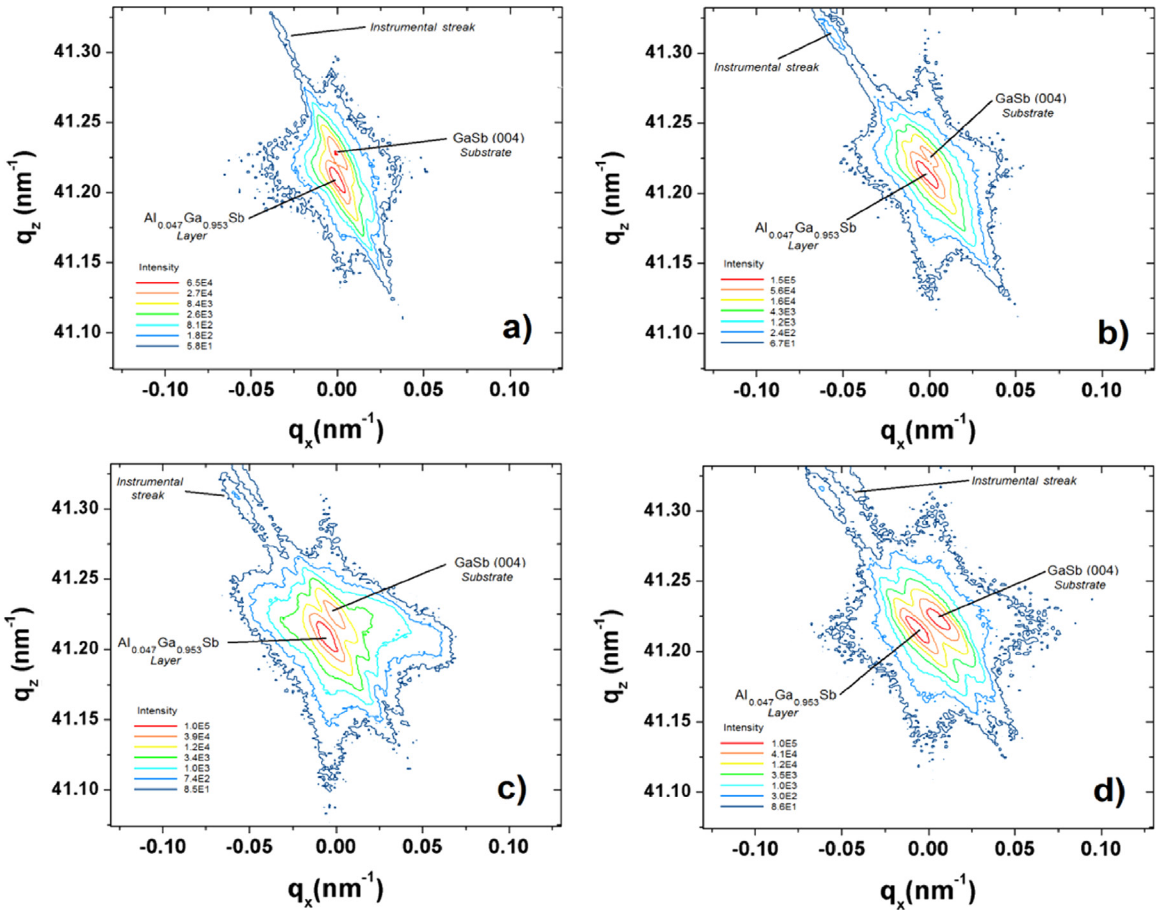


**Fig. 3.** Measured RSMs of the symmetric 004 Bragg reflection for $Al_{0.047}Ga_{0.953}/GaSb$ samples with different thickness 2.72 (a), 3.84 (b) 4.70 (c) and 5.43 μm.

$$\tan(\gamma) = \frac{\Delta q_x^{(004)}}{\frac{4}{a_s} - \left|\Delta q_z^{(004)}\right|} \tag{1}$$

The coordinates from (004) RSM lead to the calculation of epilayer out-of-plane lattice constant $a_\perp$ along [001]. Assuming as a first approximation a tetragonal unit cell, $a_\perp$ is calculated from the epilayer coordinates by [12].

$$a_\perp = a_S\left[1 - \frac{\Delta q_z^{(004)}}{\frac{4}{a_s} + \Delta q_z^{(004)}}\right] \tag{2}$$

where $a_S$ is the substrate lattice constant.

In order to find the parallel parameter $a_\parallel$ and the perpendicular parameter $a_\perp$ the asymmetric rocking curves around the 115 reflection were obtained, calculating the corresponding values using the Macrander's formulas [17] and the relaxation formula:

$$a_\perp = a_S \frac{\sin\theta_B}{\sin(\theta_B + \Delta\theta)} \frac{\cos\tau_S}{\cos(\tau_S + \Delta\tau)} \tag{4}$$

$$a_\parallel = a_S \frac{\sin\theta_B}{\sin(\theta_B + \Delta\theta)} \frac{\sin\tau_S}{\sin(\tau_S + \Delta\tau)} \tag{5}$$

$$R = \frac{a_\parallel - a_s}{a_x - a_s} \tag{6}$$

where $\theta_B$ is the Bragg angle for the (115) direction, $\tau_s$ is the angle between (115) plane and the surface plane of the sample, $_{ax}$ is the parameter of layer totally relaxed, $\Delta\theta$ and $\Delta\tau$ are obtained with the following equations [17]:

$$\Delta\theta = \frac{\Delta\omega^+ + \Delta\omega^-}{2} \tag{7}$$

$$\Delta\tau = \frac{\Delta\omega^+ - \Delta\omega^-}{2} \tag{8}$$

$\Delta\omega^+$ is the difference between substrate and layer peak in (115) direction, $\Delta\omega^-$ is the difference between the substrate and the layer peaks in $(-1-15)$ direction.

## 4. Results and discussion

Fig. 2 is shows the (004) rocking curves from the layers with different thicknesses, it can be seen that relative peak amplitude corresponding to the epitaxial layer becomes more intense as the thickness of the layer increases and its position shift to larger angles. This behavior can be attributed to layer relaxation when its thickness increases since as the layer relaxes its perpendicular parameter decreases producing a reduction in distance between the peaks corresponding to the layer and substrate. Using the (115) asymmetric rocking curves; it is possible to evaluate the parameters of the distorted cell in layers.

From the analysis of the symmetric and asymmetric HRXRD rocking curves around the (004) and (115) reflections, the Al content, the relaxation degree R, The full width at half maximum (FWHM), the parallel and perpendicular parameters of the layers were calculated and they are shown in Table 1 together the corresponding thicknesses of the layers measured in a SEM.

It can be observed that relaxation increases when the growth time, and therefore the layer's thickness, increases. This result is also supported by FWHM values of the (004) X-ray rocking curves of layers since it increases as consequence of formation of dislocations during the relaxation process. From this it is possible to conclude that thicker layers have lower crystal quality compared to others.

Fig. 3(a)–(d) shows the symmetric (004) RSMs in standard reciprocal units $q_z$ and $q_x$, from the structures formed by $Al_{0.047}Ga_{0.953}Sb$ on GaSb with different layer thicknesses. The presence of the epitaxial layer can be noticed as a region of diffraction below the substrate diffraction.

From the analysis of the symmetric and asymmetric HRXRD rocking curves around the (004) and (115) reflections, the Al content, the relaxation degree R, The full width at half maximum (FWHM), the parallel and perpendicular parameters of the layers were calculated and they are shown in Table 1 together the corresponding thicknesses of the layers measured in a SEM.

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Fig. 2(b) shows the variation of tilt and relaxation values as a function of thickness. The relaxation as a function of thickness is in agreement to Matthews and Blakeslee's mechanical equilibrium model for critical thickness [6] applied to this system. The estimated critical thickness is around 3 μm, so the layer with a thickness of 2.72 μm is the only layer without relaxation and the others layers with higher thickness are progressively relaxed.

## 5. Conclusions

The data obtained on the basis of standard equations allowed the characterization of $Al_{0.047}Ga_{0.953}Sb$ layers grown by LPE (thickness range 2–5 μm). High-resolution X-ray reciprocal space mapping has been shown to be a reliable tool for comprehensive characterization of partially relaxed $Al_{0.047}Ga_{0.953}Sb$ layers grown on GaSb (001) substrates. Based on the theory it is possible to find quantitatively, from high resolution RSM peak position the misorientation of layer respect to substrate. It is shown that both symmetric and asymmetric rocking curves contain enough information to obtain the Al content, relaxation, perpendicular and parallel parameters and lattice misfit. It was found that tilt in these layers seem to be necessary to accommodate the strain relaxation. Also, the thickness values obtained from SEM measurements are in very good agreement with the relaxation and Matthews and Blakeslee's mechanical equilibrium model for critical thickness.

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