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Critical thickness of MBE-grown $Ga_{1-x}In_xSb$ ($x < 0.2$) on GaSb

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Abstract

Several $Ga_{1-x}In_xSb$ layers, capped with 1 μm of GaSb, were grown on GaSb(001) substrates by molecular beam epitaxy in a Varian Gen II Modular system using either the conventional sample growth position with substrate rotation, or a tilted sample position with no substrate rotation. The GaInSb layers were examined by X-ray diffraction (XRD) using both symmetrical and asymmetrical reflections. The "tilted sample method" gave a variation of ± 25 % in thickness of the $Ga_{1-x}In_xSb$ layers, while the In content varied by ± 10 % around the nominal value. The disappearance of thickness fringes in 004 XRD scans was used to determine the onset of relaxation, as determining the in-plane lattice constant for tilted samples was found to be difficult. Determining residual strain in samples grown by the tilted method was likewise found to be very difficult. The critical thickness for several In mole fractions between 5 % and 19 % was determined and was found to be from 2.1 to 2.6 times higher than predicted by Matthews and Blakeslee (1974) but lower than predicted by People and Bean (1985).

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1. Introduction

Unrelaxed $Ga_{1-x}In_xSb$ epilayers grown on GaSb, or on alloys lattice-matched to GaSb, are useful for mid-infrared (MIR) emitters [1-3]. There has to the authors' knowledge, however, never been a systematic examination of the critical thickness (t_c) of $Ga_{1-x}In_xSb$ on GaSb. The closest is the work by Kuramochi *et al.* [4] where the critical thickness of $Ga_{1-x}In_xSb$ on $Al_{0.35}Ga_{0.65}Sb$ was partly mapped using photoluminescence (PL) emission from single quantum wells. Practical uses of strained $Ga_{1-x}In_xSb$ layers would be as an active layer buried beneath a thick layer (> 1 μm) lattice-matched to GaSb. For this reason, we have chosen to study $Ga_{1-x}In_xSb$ layers capped with around 1 μm of GaSb.
In this work, we present the first results of t_c for GaInSb with an In mole fraction between 5 % and 19 % and make use of the tilted sample method (TSM) for determining the critical thickness. The tilted sample method offers a way of obtaining a range of thicknesses and compositions on a single wafer. This allows a determination of critical thickness using a single sample.

2. Experimental procedure

Several $Ga_{1-x}In_xSb$ layers were grown on Te-doped GaSb(001) 2" quarter wafer substrates in a Varian Gen II Modular molecular beam epitaxy (MBE) system. One set of layers was grown conventionally with substrate rotation while another set was grown with a method utilizing tilted samples akin to the method used by Fox *et al.* [5]. Fig. 1 and Fig. 2 show sketches of the growth chamber source positions and sample position. The Ga source is a dual filament furnace, while the In source is a SUMO furnace and the Sb source is a valved cracker furnace.

The structure grown conventionally consisted of a 1.9 μm thick Te-doped GaSb buffer followed by an n-p junction (using Te and Be as dopants) in a $Ga_{1-x}In_xSb$ layer with an In mole fraction around 8.5 % (as determined from X-ray diffraction (XRD) measurements). The structure was then capped with a 1 μm thick Be-doped GaSb layer. The in-plane and out of plane lattice constants of the $Ga_{1-x}In_xSb$ layer were extracted using the refinement technique described by Fatemi [6] from measurements with a Philips PW 1880 X-ray diffractometer.

Using the TSM, samples were grown without rotation and with the sample tilted downwards about 60° from the normal position, expected to result in gradients of both Ga and In growth rates across the sample. The average thicknesses of the layers were tentatively the same as for the structure described above, but the layers were not doped. Variations in GaSb and InSb growth rates for tilted samples were calibrated by growing a GaSb/GaInSb superlattice (SL) with the TSM and using *ex-situ* XRD measurements. The layers of the tilted samples were examined with a Bruker D8 Discover XRD system using both symmetric and asymmetric reflections. Asymmetric measurements (using 115, 117, 226, and 335 reflections) were carried out using a modified zone technique [7, 8]. A 1 mm diameter circular aperture was placed in front of the XRD source (and also in front of the detector when measuring the samples with lowest In content). Simulations on samples grown using TSM was performed on the LEPTOS software from Bruker. In order to find the area of a sample grown using the TSM where the sample had relaxed, a manual binary search was performed, after which a rectangular area was mapped with XRD scan points 1.5 to 7 mm apart. The step size used in each case was determined by the area to be covered and time considerations. For all samples, the In containing layers and the first 10 nm of the following GaSb layer were grown at 450°C, as measured by a pyrometer. The top and bottom GaSb layers were grown at 490°C. In the case of the GaSb/GaInSb SL, the whole structure was grown at 450°C.

3. Results and discussion

Fig. 3 and Fig. 4 show the thickness and the indium content distribution, respectively, obtained across a sample grown by the TSM. The data were obtained from an unrelaxed control sample using 004 XRD scans and simulations. A thickness variation of ±25 % and an indium content variation of ±10 % were obtained. The ranges for the variation in GaInSb layer thickness and In content across the samples were found to be similar for several samples, but the areal distribution pattern was somewhat different from sample to sample. We believe these differences to be caused by slightly different azimuthal angles of the wafers during growth. Due to the problems in reproducing the same thickness/In content pattern over several SL samples, such samples could not be used to determine the thickness and In content in the MIR structures. Instead, the In content and layer thickness were determined from the XRD scans from each GaSb/GaInSb/GaSb MIR structure using simulations. Uncertainties in thickness and In content values obtained from the simulation on unrelaxed samples have been estimated to ± 1 nm and ± 0.001, respectively, for an In fraction of 0.082.

As can be seen from Fig. 5, the XRD thickness fringes from the $Ga_{1-x}In_xSb$ layer dampens and disappears when going from position (a) to (c) on the sample, while simulated thickness increases from 71.5 nm at position (a) to 73.4 nm at (b) and 74.1 nm at (c). The In content was 12.3 % in positions (a) and (b) and 12.4 % in position (c). We believe this is due to the onset of relaxation of the In containing layer. Selvig *et al.* [9] and Chen *et al.* [10] have found that the fringes disappear after the onset of relaxation. For the determination of relaxation, scan (a) would be considered to arise from unrelaxed GaInSb, whereas (c) would be considered to be from partially relaxed GaInSb. Since the GaInSb layer has started to relax at position (c), the In content at this position obtained from simulations will no longer necessarily be accurate, as the simulation assumes a fully strained layer, but will be used here although it is an approximation. Looking at the thickness and In content values from (a) to (c) in Fig. 5, it seems to be a fairly good approximation. It should be noted that this measurement method does not measure at which thickness dislocations start forming due to strain but rather at which thickness enough dislocations have formed so that the XRD is affected. However, Chen *et. al.* [10] found that this is also the point when the PL from the material starts deteriorating. As applications for this material system is mostly light emitters, the error made in setting t_c at the thickness where it can be detected by XRD should not affect its usefulness.

Verification of the relaxation by examining the in-plane lattice constant of the $Ga_{1-x}In_xSb$ layer using asymmetrical reflections was attempted, but the results were inconclusive. We believe the reason for this is degradation in the accuracy of the asymmetrical measurements due to the varying In content and thickness of epilayers of the sample grown by the TSM, and the fact that the change in the in-plane lattice constant is small at the onset of relaxation.

Samples grown with the tilted sample method have to be examined with a small X-ray spot size of 1 mm in order to minimize the variations in thickness and In content across the area sampled by the XRD. Even so, there will still be variations across the sampled area. The X-ray beam will have an elliptical spot on the sample surface with minor and major axis lengths of 1 mm and 2 mm, respectively, for the 004 GaSb reflection. However, for

some reflections used in asymmetrical measurements the spot on the sample surface will be much larger due to the low angle of incidence of the X-ray beam. In our case, the 335 reflection was worst in this respect with a 3.7 mm diameter spot. For the Bruker XRD, the reduction in signal caused by the aperture led to a large increase in integration time compared to uniform samples. These two problems combined made it very difficult to do accurate asymmetrical measurements on samples grown using the tilted sample method.
The fact that we have no accurate way of determining the thickness when the sample has started to relax and thickness fringes has disappeared, and that the asymmetrical measurements required to determine the In content accurately are difficult, makes the extraction of residual strain from the tilted samples very difficult.
Full width at half maximum (FWHM) of In containing layers were compared to the FWHM of the simulated spectra and were found to be slightly higher for the unrelaxed layers. When comparing the FWHM of scans where the thickness fringes had just disappeared to scans where the thickness fringes were still visible, we found that the FWHM in some scans had increased whereas in others it had decreased. The FWHM of the simulated curves also oscillated with changing thickness, but the trend was toward lower FWHM for thicker layers. Measurements of the FWHM of unrelaxed samples exhibited the same behavior. Both the difference between simulated and measured FWHM and the change in FWHM after thickness fringes disappeared were within ± 10 %. For layers significantly thicker than t_c, the FWHM was found to increase as expected from a layer going through relaxation. The simulations performed assume a uniform layer thickness and In content. The difference between simulations and the measured XRD scans are most likely due to the variations in thickness and In content over the area probed by the XRD beam.
Fig. 6 shows the data obtained by the TSM and the conventional method. The data around 8.5 % In obtained using the conventional method are consistent with the data which were obtained using the TSM. The data for the fully strained layers are consistent with previous work [4]. The People and Bean (PB) [11] theory for critical thickness predicts the onset of misfit dislocations while assuming that there are no existing threading dislocations, whereas Matthews and Blakeslee (MB) [12] predicts the thickness at which existing threading dislocations will cause misfit dislocations. As noted before, the measurement method used in this work does not measure at which thickness dislocations start forming due to strain but rather at which thickness enough dislocations have formed so that the XRD is affected. Further XRD investigations using the conventional method with a larger number of samples are needed to determine the difference between these two thicknesses. However, it still seems useful to compare our data to the critical thicknesses predicted by MB and PB.
From our data, we find that for 5.0 % In the t_c = 236 ± 16 nm (2.7 times larger than predicted by MB), for 12.3 % In the t_c = 73.5 ± 3 nm (2.6 times larger than MB), for 14.2 % In the t_c = 64.5 ± 1.5 nm (2.7 times larger than MB), and for 18.7 % In the t_c = 36.2 ± 2 nm (2.2 times larger than MB).

4. Conclusions

Several $Ga_{1-x}In_xSb$ layers with different In mole fractions were grown by MBE using either the conventional sample position or a tilted sample position for the purpose of determining critical thicknesses (t_c). The tilted sample method was found to be well suited to determine t_c by allowing a range of thicknesses and In mole fractions to be examined on one sample, thus reducing the number of samples required. However, using the tilted sample requires a number of longer XRD measurements per sample and makes the use of asymmetrical measurements to determine the in-plane lattice constant difficult. Furthermore, the tilted samples are unsuitable for residual strain measurements unless the thickness and In content distributions can be reproduced accurately from sample to sample. The critical thickness was found for several In mole fractions between 5 to 19 % and was found to be about 2.5 times of what MB [12] predicts but lower than predicted by PB [11].

Acknowledgements

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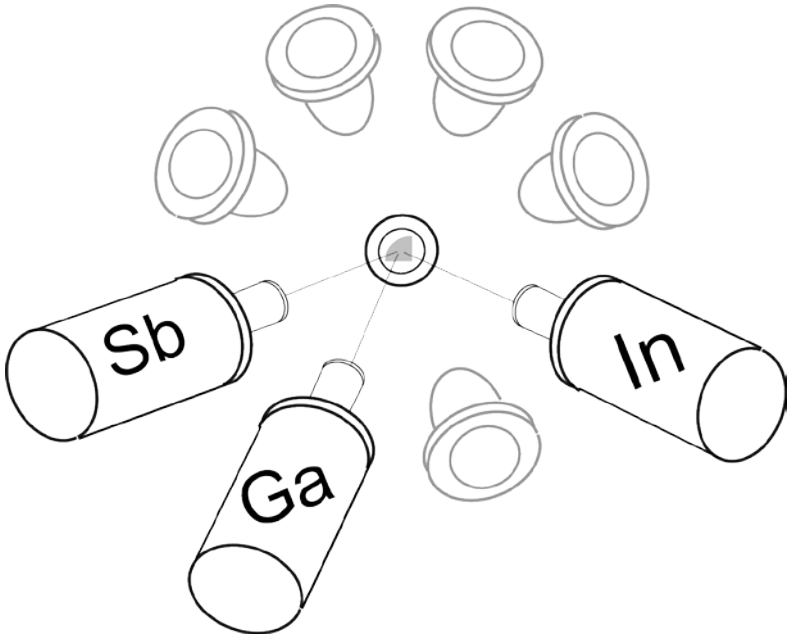

Fig. 1. Sketch of source positions in the MBE growth chamber.

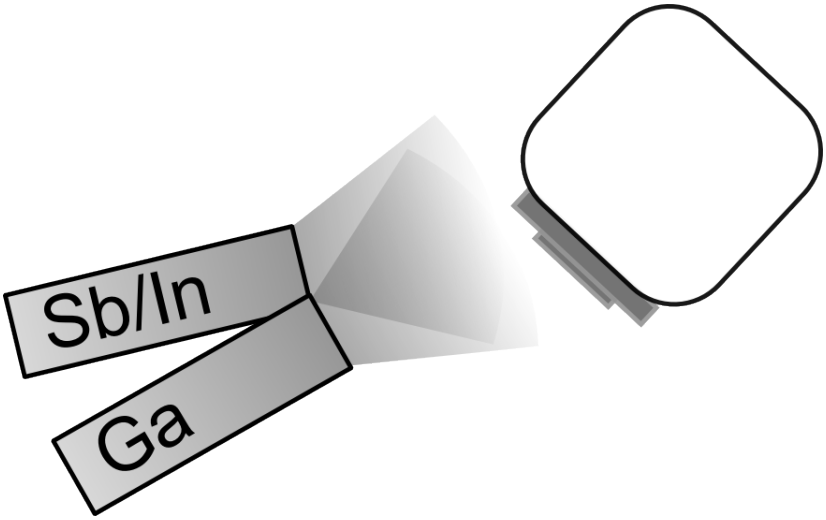

Fig. 2. Sketch of source positions and sample tilt of the “tilted sample method”.

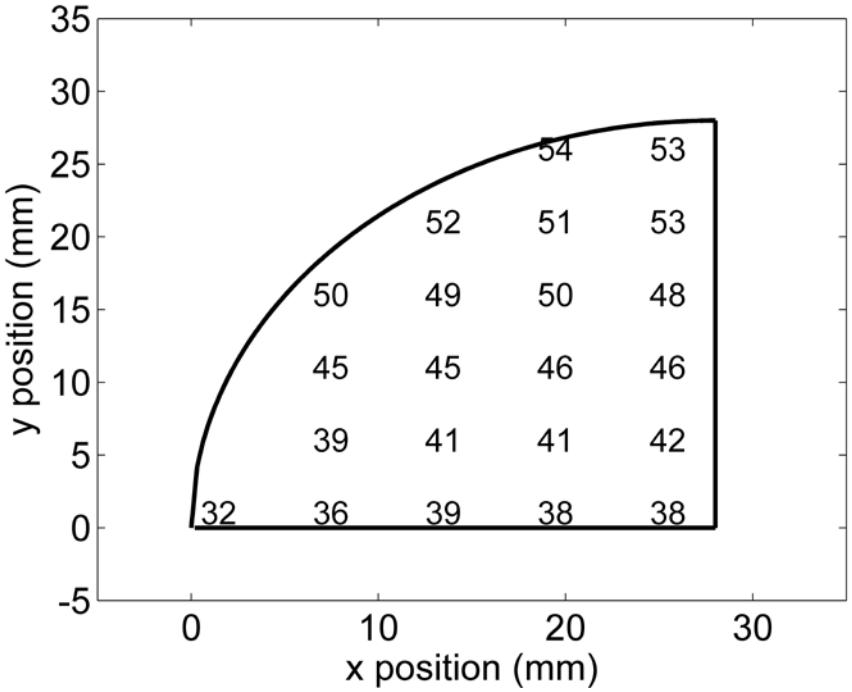

Fig. 3. Thickness (nm) of the $Ga_{1-x}In_xSb$ layer vs. position (mm) for films grown on a tilted 2" quarter wafer without rotation. No relaxation was observed in this sample (Sb 128-2).

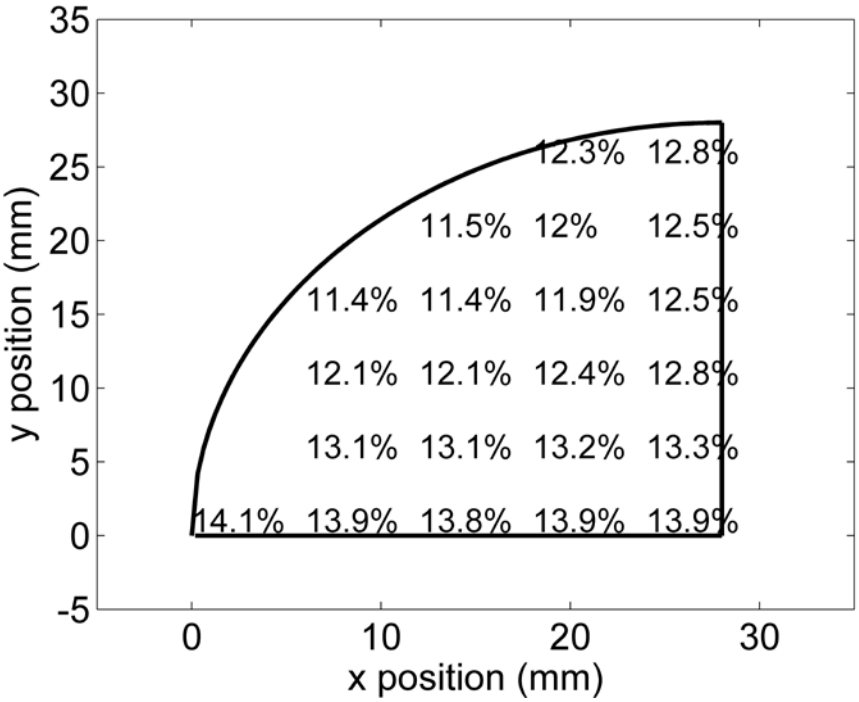

Fig. 4. In content of the $Ga_{1-x}In_xSb$ layer vs. position (mm) for films grown on a tilted 2" quarter wafer without rotation. No relaxation was observed in this sample (Sb 128-2).

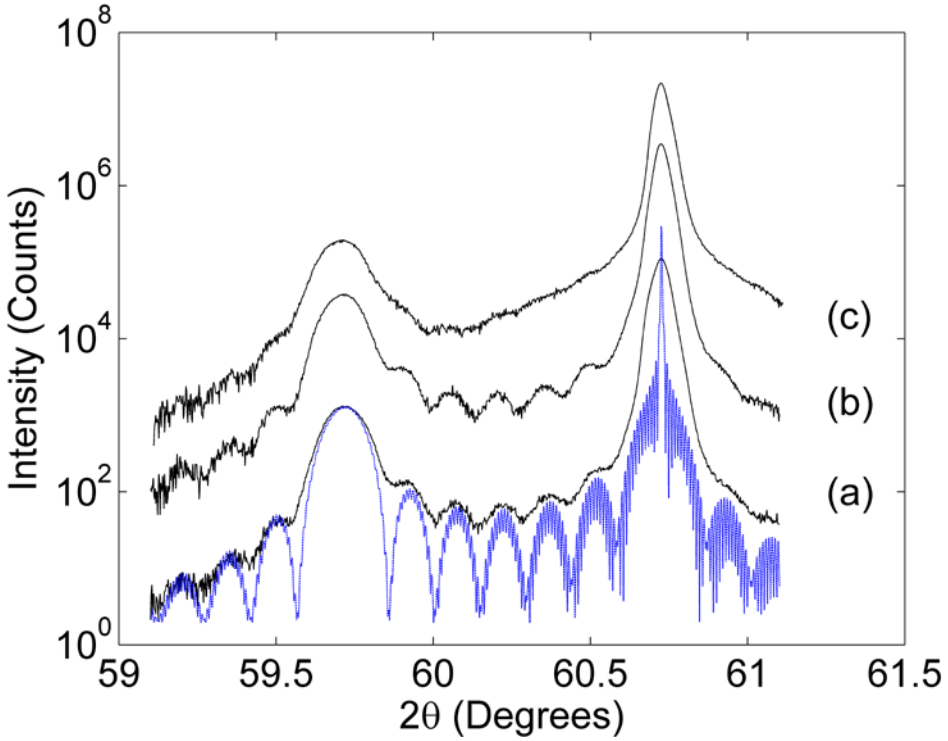

Fig. 5. 004 XRD scans from different positions on the same sample (Sb 128-3) along with a simulated spectrum for position (a). From simulations, GaInSb thickness and In mole fraction were found to be 71.5 nm and 12.3 % for (a), 73.4 nm and 12.3 % for (b), and 74.1 nm and 12.4 % for (c). The positions were 1.5 mm apart in a straight line on the sample. The XRD scans are offset vertically for clarity.

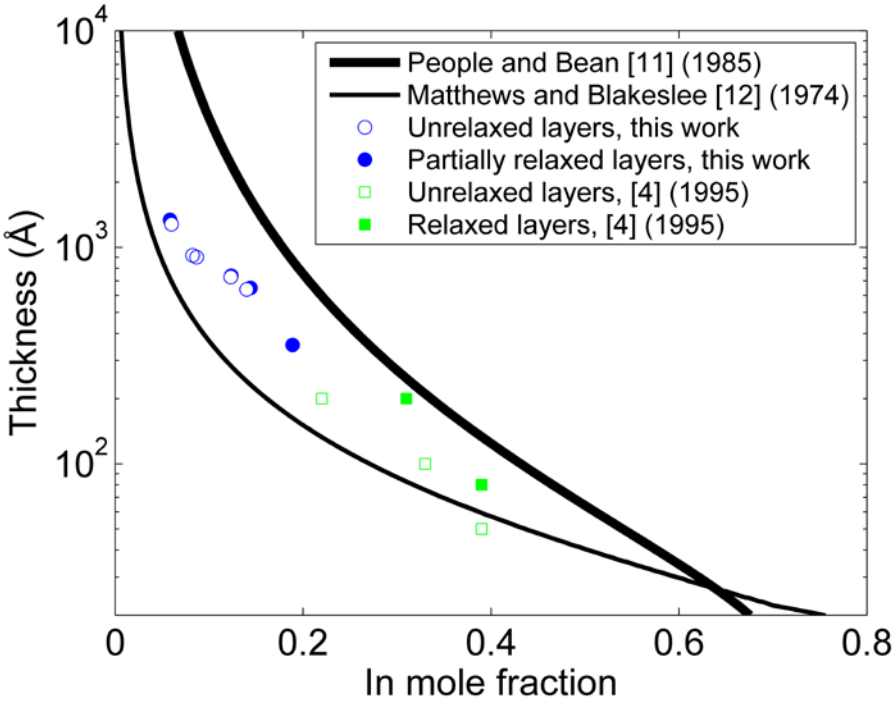

Fig. 6. Data from this work and previous work plotted along with the critical thickness at 450°C of GaSb-capped GaInSb on GaSb predicted by the Matthews and Blakeslee (1974) and People and Bean (1985) theories.