High Resolution X-ray Diffraction and X-ray Reflectivity Studies of InAs/AlGaAsSb
Deep Quantum Wells

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Abstract

High resolution x-ray diffraction (HRXRD) and grazing incidence x-ray reflectivity (GIXR) methods are powerful tools to characterize multi-layer epitaxial films. This study employs these techniques to investigate the structural properties of InAs/AlGaAsSb deep quantum wells (InAs QWs) grown by molecular beam epitaxy (MBE). Crystallinity of the AlGaAsSb layer and its interface roughness with the InAs QWs obtained from these x-ray studies were correlated with the electron mobility of InAs QWs. Subsequently the effect of its crystallinity and interface roughness to the electron mobility of the InAs QWs was clarified.

Introduction

High resolution x-ray diffraction (HRXRD) methods had extensively been employed for probing crystallinity of epitaxial films.1,2 Recently, grazing incidence x-ray reflectivity (GIXR) had been a valuable technique to investigate layer thickness, density and interface roughness of thin films.3,4 The InAs/AlGaAsSb material system has been attractive to various device applications due to conduction band offset of ~1.3eV and high electron mobility in InAs. Under optimum grown conditions, InAs/AlGaAsSb deep quantum wells could achieve very high electron mobility of 32,000 cm²/Vs at room temperature.5,6

One of the main purpose of this paper is to explore the relationship between structural properties including interface roughness and electronic properties for this InAs/AlGaAsSb system. Results on the crystallinity of AlGaAsSb bottom barrier layer as determined by HRXRD method, together with interface roughness data of AlGaAsSb/InAs obtained by GIXR method, are presented and subsequently correlated with the electron mobility of the deep quantum wells. Surface roughness data obtained from GIXR technique with computer simulation data are compared to results determined by Atomic Force Microscopy (AFM).
'This document was presented at the Denver X-ray Conference (DXC) on Applications of X-ray Analysis.

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Experimental

All samples were grown on semi-insulating (100) GaAs substrate in a specially modified VG Semicon V100 molecular beam epitaxy (MBE) system, equipped with elemental group III and group V sources, the latter producing As$_4$ and Sb$_4$ beams. Fig. 1 shows a schematic cross section and energy band diagram of InAs/Al$_{0.5}$Ga$_{0.5}$AsSb QWs.

<table>
<thead>
<tr>
<th>GaAsSb 5nm</th>
<th>AlGaAsSb 10nm</th>
<th>InAs 15nm</th>
<th>AlGaAsSb 600nm</th>
<th>GaAs Buffer</th>
<th>GaAs Substrate</th>
</tr>
</thead>
</table>

Fig. 1 Schematic cross section and energy band diagram of InAs/Al$_{0.5}$Ga$_{0.5}$AsSb QWs

energy band diagram of InAs deep quantum well structures (InAs QWs). The structure of InAs QWs consisted of 600 nm of Al$_{0.5}$Ga$_{0.5}$AsSb bottom barrier, 15 nm of InAs well, 10 nm of AlGaAsSb top barrier and 5 nm of GaAsSb cap layer. The InAs well was sandwiched between quaternary AlGaAsSb barrier layers that were lattice matched to the InAs wells.

A Philips MRD X-ray diffractometer was used for the diffraction measurement. High incident angle diffraction experiments were measured using a slit-collimated parallel beam optics, pictured schematically in Fig. 2. Horizontal angular divergence of the incident beam and angular acceptance of the diffracted beam was 0.3 deg. Azimuthal orientations of the InAs QWs were determined by (CuK$\alpha$ radiation) symmetric scans and their in-plain orientations were measured by asymmetric scans using a four-circle goniometer. Rocking curves of AlGaAsSb bottom layer were measured using Ge (220) four crystal incident monochrometer setting to isolate CuK$\alpha_1$ radiation, pictured schematically in Fig. 3. An incident beam angular divergence of 12 seconds of arc and a wavelength spread of $1.4 \times 10^{-4}$ were normally used in this study.

The measurements of GIXR were performed using a 2KW x-ray generator (RIGAKU) with a copper target. The x-ray from the copper target were collimated and monochromated by a Ge (111) single crystal. The first slit ($50 \mu$m width) allowed the CuK$\alpha_1$ line to impinges onto the sample only, and the second receiving slit was $100 \mu$m wide to restrict the intensity of the scattered radiation. A schematic diagram of the reflectometer is shown in Fig. 4. The reflectivity curves were taken in symmetrical $2\theta$-$\theta$ scans. Extreme care was
taken to optimize the alignment of specimen to obtain as high resolution as possible. The accuracy of angular measurement was 0.001 deg for the reflectivity measurement. The step-size was 0.002 deg with a scan time of 4 seconds per data point.

![A schematic diagram of the slit-collimated parallel beam optics](image1)

**Fig. 2** A schematic diagram of the slit-collimated parallel beam optics

![A schematic diagram of the 4-crystal Ge(220) incident monochromometer optics](image2)

**Fig. 3.** A schematic diagram of the 4-crystal Ge(220) incident monochromometer optics

![A schematic diagram of the X-ray reflectivity measurement](image3)

**Fig. 4** A schematic diagram of the X-ray reflectivity measurement

The measured reflectivity was analyzed by using Parratt's recursion formula\textsuperscript{6,7} modified by the distorted wave Born approximation for surface and interface roughness\textsuperscript{8} and to Marquard method for parameter (layer thickness, surface and interface roughness) optimization. The interface roughness was expressed by using an exponential factor to the reflection coefficient.

Electric characteristics of the samples were measured Hall effect using the conventional van der Pauw method at room temperature.
Results

1. Orientation relationships

High incident angle x-ray diffraction patterns for the InAs layer, AlGaAsSb bottom layer and GaAs substrate were measured using a 4-crystal Ge(220) incident monochrometer setting with 0.45mm slit in front of the detector. Typical $2\theta/\omega$ scan profile is shown in Fig. 5. The lattice constant of the InAs well was find to be almost the same as that of the bulk. Here, $\omega$ is the angle between the incident x-ray and sample surface.

![Fig. 5 2 $\theta/\omega$ scan profile of InAs-QWs](image)

$\phi$ scans of GaAs(511) plane, AlGaAsSb(511) plane and InAs(511) plane were performed to determine this orientation relationships with respect to each other as shown in Fig. 6. This figure clearly shows that the in-plane orientation of the AlGaAsSb $\langle 100 \rangle$ axis parallels to GaAs $\langle 100 \rangle$ axis as well as to the InAs $\langle 100 \rangle$ axis. The film/substrate orientation relationship as determined by $\phi$ scans is shown schematically in Fig. 7.

![Fig. 6 $\phi$ scan profiles of GaAs(511), AlGaAsSb(511), and InAs(511)](image)
2. Effects of As$_4$/Sb$_4$ flux ratio to crystallinity and electron mobility

The As$_4$/Sb$_4$ flux ratio for the growth of AlGaAsSb buffer layer was found affecting the electron mobility in the InAs well in a manner as shown in Fig. 8. The crystallinity of AlGaAsSb bottom layers were evaluated by measuring rocking curves of AlGaAsSb(400) using Ge(220) four crystal incident monochrometer setting. Rocking curves for these AlGaAsSb buffer layers grown under various As$_4$/Sb$_4$ flux ratio are shown in Fig. 9. Full width at half maximum (FWHM) of these rocking curves were calculated and correlated with the electron mobility of the deep quantum wells as shown in Fig. 10.
3. Effects of As$_4$/In flux ratio to interface roughness and electron mobility

In Fig. 11, we show the As$_4$/In beam ratio dependence of electron mobility during the growth of InAs layer. It is seen that the electron mobility increases with decreasing the As$_4$/In beam ratio. Due to the difficulty for obtaining double axis x-ray rocking curves from thin InAs layers of about 15 nm thick, GIXR technique was performed.

The reflectivity was analyzed by using Parratt’s recursion relation$^{7,8}$:

$$R_{n-1,n} = a_{n-1} \left\{ \frac{R_{n,n+1} + F_{n-1,n}}{R_{n,n+1} F_{n-1,n} + 1} \right\}$$  \hspace{1cm} (1)

where

$$R_{n,n+1} = \frac{\gamma E_n^R}{E_n}$$, \hspace{0.5cm} a_n = \exp \left( \frac{i \pi}{\lambda} \phi_n d_n \right), a_0 = 1$$

and

$$\phi_n = \left( \theta^2 - 2\delta_n - i2\beta_n \right)^{1/2}$$

In Eq. (1), the parameter $a_n$ is an amplitude factor, and $E_n$ describes the incident electric vector amplitude in the n-th layer and $E_n^R$ describes the reflected amplitude in the layer. $F_{n-1,n}$ is the coefficient for reflection and $\theta$ is the incident angle. The measured intensity can
be obtained by the ratio of the incident and reflected beams\(^7,8\) :

\[
|R_{0,1}(\theta)|^2 = \left| \frac{E^R_1}{E_1} \right| \quad (2)
\]

For surface and interface roughness, \(F_{n-1,n}\) is modified as follows\(^9\) :

\[
F_{n-1,n} = F_{n-1,n} \exp \left\{ -1/2 \left( \frac{\sigma_{n-1,n}^2 \lambda^2}{2} \phi_{n-1} \phi_n \right) \right\}
\]

\[
= \frac{\phi_{n-1} - \phi_n}{\phi_{n-1} + \phi_n} \exp \left\{ -1/2 \left( \frac{\sigma_{n-1,n}^2 \lambda^2}{2} \phi_{n-1} \phi_n \right) \right\}
\]

where \(\sigma_{n-1,n}\) is the root-mean-square value of the roughness of the \(n-1\) \(\rightarrow\) \(n\) interface.

In Fig. 12, the experimental data of the specular reflected intensity (dot) obtained from GIXR together with the computer simulation (solid line) for two samples grown under different As\(_4/\)In flux ratio conditions are shown.

![Fig. 12 Specular reflected intensity obtained from GIXS (dot) and its computer simulation (solid line) for two samples grown under different As\(_4/\)In flux ratios](image)

**2 \(\theta\) (deg)**

Fig. 12 Specular reflected intensity obtained from GIXS (dot) and its computer simulation (solid line) for two samples grown under different As\(_4/\)In flux ratios

**Interface roughness**

<table>
<thead>
<tr>
<th></th>
<th>H115</th>
<th>H063</th>
</tr>
</thead>
<tbody>
<tr>
<td>GaAsSb</td>
<td>1.3</td>
<td>1.7nm</td>
</tr>
<tr>
<td>AlGaAsSb</td>
<td>1.9</td>
<td>1.5nm</td>
</tr>
<tr>
<td>In(_\lambda)S</td>
<td>1.2</td>
<td>2.1nm</td>
</tr>
<tr>
<td>AlGaAsSb</td>
<td>1.0</td>
<td>1.3nm</td>
</tr>
</tbody>
</table>

Fig. 13 Interface roughness obtained by simulation
It is seen in this figure that the reflected intensity of the sample (H063) grown using a As/In flux ratio of 195 decreased more rapidly than that of the sample (H115) grown using a As/In flux ratio of 100. The interface roughness as predicted by computer simulation shows clearly that sample: H063 had a larger surface and interface roughness than that of sample: H115 (Fig. 13).

Furthermore, we have analyzed all samples and obtained the surface and interface roughness of each layer as shown in Table 1. The computer simulation data for the surface roughness was compared with the AFM data and good agreement was obtained, showing good validity of this simulation data.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Mobility (cm²/Vs)</th>
<th>H115</th>
<th>H179</th>
<th>H064</th>
<th>H063</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>31500</td>
<td>27370</td>
<td>19700</td>
<td>15400</td>
</tr>
<tr>
<td>surface</td>
<td>(by AFM)</td>
<td>1.3</td>
<td>1.4</td>
<td>1.5</td>
<td>1.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(1.7)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>between GaAsSb and AlGaAsSb</td>
<td>1.9</td>
<td>1.0</td>
<td>1.3</td>
<td>1.5</td>
<td></td>
</tr>
<tr>
<td>between AlGaAsSb and InAs</td>
<td>1.2</td>
<td>1.5</td>
<td>1.6</td>
<td>2.1</td>
<td></td>
</tr>
<tr>
<td>between InAs and AlGaAsSb(b)</td>
<td>1.0</td>
<td>1.1</td>
<td>1.0</td>
<td>1.3</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Surface and interface roughness for all samples predicted by the computer simulation. Note that the AFM data agrees very well with this simulation results (unit: nm)

Computer simulation result using a wide range of roughness (1.0-1.3 nm) for the bottom AlGaAsSb/InAs interface indicated no appreciable effect to the calculated reflectivity spectrum. However, the simulated reflectivity spectrum was found sensitive to the upper AlGaAsSb/InAs interface roughness. Fig. 14 shows a remarkable relationship between with the upper interface roughness. The electron mobility is seen increasing dramatically with decreasing this interface roughness. This result leads to the conclusion that strong relationship between the upper interface roughness and electron mobility of the InAs DQWs exists in this system.

Figure 14. The relationship between the electron mobility in the InAs well with the InAs/AlGaAsSb bottom interface roughness
Conclusion

We have characterized the structures and electronic properties of InAs deep quantum wells using high resolution XRD and grazing incident x-ray reflectivity. By high resolution XRD measurement, we could clarify the As/Sb beam flux ratio effect during the growth of AlGaAsSb bottom buffer layer to the crystallinity, and then to the electron mobility in the InAs well. X-ray reflectivity measurement together with computer simulation further indicates that InAs/AlGaAsSb bottom interface roughness impose an influence on the electron mobility in InAs well. This roughness can be reduced to a minimum by the choice of a proper As/In flux ratio during the growth of InAs layer. In this way, by using HRXRD and GIXR methods, epitaxial thin films as InAs Qws could be well characterized.

Acknowledgments

We would like to thank Mrs. Yoshida for technical assistance of AFM measurement. Thanks are due to Dr. T. Ikegami, Mr. S. Ibe, Dr. T. Neki and Mr. T. Konishi of our laboratories for continuous encouragement. Scincere thanks are also due to Dr. Peter Yuen for reading the manuscript.

References

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