THE OPTIMIZATION OF INTERFACES IN InAsSb/InGaAs STRAINED-LAYER SUPERLATTICES GROWN BY METAL-ORGANIC CHEMICAL VAPOR DEPOSITION

CONF-940411--9

R. M. Biefeld, K. C. Baucom, and S. R. Kurtz
Sandia National Laboratory, Albuquerque, NM

ABSTRACT

We have prepared InAsSb/InGaAs strained-layer superlattice (SLS) semiconductors by metal-organic chemical vapor deposition (MOCVD) under a variety of conditions. The presence of an InGaAsSb interface layer is indicated by the x-ray diffraction patterns. The optimized growth conditions involved the use of low pressure, short purge times, and no reactant flow during the purges. MOCVD has been used to prepare an optically pumped, single heterostructure InAsSb/InGaAs SLS / InPSb laser which emitted at 3.9 μm with a maximum operating temperature of approximately 100 K.

INTRODUCTION

We are exploring the growth of InAs_{1-x}Sb_x/In_{1-y}Ga_yAs SLS's for their possible use in mid-wave, 2-5 μm infrared optoelectronic and heterojunction devices. This system was chosen because the compositions span the 2-5 μm wavelength range and they can be grown lattice matched to InAs with type I band offsets. Recent work by Menna et al. on a metal-organic chemical vapor deposition (MOCVD) grown 3.06 μm diode laser yielded a maximum operating temperature of 35 K and threshold current densities of 200 - 330 A/cm². These results indicate the potential and the need for devices operating in this wavelength range.¹ Our previous studies in the Sb rich end of the InAsSb ternary system have demonstrated accurate composition control through the use of a thermodynamic model. We have made high quality infrared detectors in our laboratory from doped strained-layer superlattices (SLS's) grown by MOCVD in the Sb rich end of this ternary.^{2,3}

Our previous work in the As rich end of these same materials focused on the growth of InAs_{1-x}Sb_x/In_{1-y}Ga_yAs heterostructures on InAs.^{4,5} These studies concentrated on the growth and characterization of the InAsSb layers due to the importance of this material in the active devices. Detailed growth conditions for InAs_{1-x}Sb_x/In_{1-y}Ga_yAs SLS's and InAs_{1-x}Sb_x alloys, and the growth and characterization of an 4 μm infrared photodiode were reported.

In this work, a series of experiments was carried out to determine the effects of growth conditions, purge times, and reactant flow sequences on the x-ray diffraction patterns of these SLS's. Herein we also report on an optically pumped laser stripe constructed from these strained heterostructures. The details of the growth conditions and characterization results and the applicability to infrared devices of As rich InAs_{1-x}Sb_x materials are discussed.

EXPERIMENTAL

This investigation was carried out in a previously described horizontal MOCVD system.⁶ The sources of In, Sb and As were trimethylindium (TMIn), trimethylantimony (TMSb), trimethylgallium (TMG), and 100 percent arsine (AsH₃), respectively. Hydrogen was used as the carrier gas at a total flow of 6 SLM. The III/V ratio was varied from 2.7 to 10.3 over a temperature range of 475-525 °C, at total growth pressures of 200 to 660 Torr and growth rates of 0.75 to 3.0 $\mu\text{m}/\text{h}$.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Figure 1. The upper curve is the observed x-ray diffraction pattern of the (004) reflection of CVD1263 compared to a calculated pattern in the lower curve to illustrate the broadness of the observed peaks and their asymmetry.

The growth was performed on (001) InAs substrates. Diodes have been prepared using Zn as the p-type dopant with diethylzinc as the precursor and undoped SLS as the n-type material. InAs was cleaned by degreasing in solvents and deionized water. It was then etched for 20 to 30 seconds in a 10:1 mixture of sulfuric acid and hydrogen peroxide, rinsed with deionized water and blown dry with nitrogen.

Sb compositions, x , reported for the $\text{InAs}_{1-x}\text{Sb}_x$ layers were determined by x-ray diffraction using a Cu x-ray source and a four crystal Si monochromator. The (004) reflection was used to measure the lattice constant normal to the growth plane and the (115) or (335) reflections were used to determine the in-plane lattice constant.⁶ In this way the composition determination is corrected for partial strain relaxation by misfit dislocations. The simulations were done using the Bede RADS program.

Infrared photoluminescence was measured at 14 K using a double-modulation technique which provides high sensitivity, reduces sample heating, and eliminates the blackbody background from infrared emission spectra.⁷ Laser emission samples were made from ≈ 100 μm long cleaved stripes. The stripes were pumped with a Ti-sapphire laser operating at 825 nm, with 30 nsec pulses and a 1 kHz repetition rate. The pump light was focused onto the top of the stripe using a cylindrical lens. To heat-sink the stripes, they were pressed into indium with a sapphire disc. In this configuration, the laser output is reflected from the indium, and optical collection of the laser emission is inefficient. Laser emission was measured with a 0.25 m monochromator and an InSb detector. The absorption measurements were carried out on polished samples using a Fourier transform infrared spectrometer.

Figure 2. The upper curve is the observed x-ray diffraction pattern of the (004) reflection of CVD1263 compared to a calculated pattern in the lower curve to illustrate the improved match when interface layers are included in the x-ray simulation.

RESULTS AND DISCUSSION

An x-ray diffraction pattern of the (004) reflection of a selected $\text{InAs}_{1-x}\text{Sb}_x/\text{In}_{1-y}\text{Ga}_y\text{As}$ SLS is shown in Figure 1. The pattern shown is for sample CVD1263. This sample was grown at atmospheric pressure with 30 second purges between layers with uninterrupted AsH_3 flow. The calculated pattern is for an $\text{InAs}_{0.9}\text{Sb}_{0.1}/\text{In}_{0.91}\text{Ga}_{0.09}\text{As}$ SLS with 100 Å/130 Å layer thicknesses. The slight asymmetry and the width of the peaks is most likely due to the existence of an interface between the layers. Several simulations were tried in an effort to match the observed spectrum. If the thickness and composition of both the InGaAs and InAsSb layers were varied then the pattern became uniformly broad. If only the Sb composition and layer thickness was varied in the InAsSb layer but the sum of the thickness of both layers remained constant then the pattern remained symmetric and sharp. If the compositions of the layers including two $\text{In}_{1-x}\text{Ga}_x\text{As}_{1-y}\text{Sb}_y$ interface layers were varied but the total period remained constant the simulation produced uniformly broadened peaks. The best match was obtained by varying the composition and thickness of the InAsSb layer and the interface layers but not for the InGaAs layer. This simulation is illustrated in Figure 2.

In Figure 2 the calculated pattern is for a structure that consists of 100 layers grouped in five segments with different interface layer compositions and thicknesses. The structure contains the basic period of $\text{In}_{1-x}\text{Ga}_x\text{As}_{1-y}\text{Sb}_y / \text{InAs}_{1-z}\text{Sb}_z / \text{In}_{1-x}\text{Ga}_x\text{As}_{1-y}\text{Sb}_y / \text{In}_{1-w}\text{Ga}_w\text{As}$, where the interface layers are identical. The $\text{In}_{1-w}\text{Ga}_w\text{As}$ layers are invariant throughout the structure with compositions of $w = 0.09$, and thicknesses of 110 Å each. The thickness of the $\text{InAs}_{1-z}\text{Sb}_z$ layers are all equal but the composition varies from $z = 0.06$ to 0.08 in 4 steps of 0.005. Both the composition and the thickness of the interface layers change with the layer thickness varying from 2 to 10 Å in steps of 2 Å and the Sb composition

Figure 3. The upper curve is the observed x-ray diffraction pattern of the (004) reflection of CVD1479 compared to a calculated pattern in the lower curve to illustrate the improved diffraction pattern obtained using the optimum growth conditions.

changing from $z = 0.05$ to 0.09 while the Ga composition remains unchanged at $x = 0.1$. The order of the layers makes no difference to the calculated pattern. This simulated pattern in Figure 2 is a good match to the observed pattern and is support for the existence of an interface layer as the explanation for the width and asymmetry observed in the x-ray diffraction pattern.

A variety of growth conditions were explored in an effort to improve the sharpness of the x-ray diffraction pattern. The growth temperature, growth rate, and V/III ratio were held constant while the growth interrupts were varied at both atmospheric and low pressure (630 and 200 Torr). The optimum x-ray diffraction pattern was obtained at low pressure with a 10 second growth interrupt and no reactants flowing during the interrupt. An x-ray diffraction pattern of an SLS grown using the optimum growth technique is shown in Figure 3. The simulated pattern in Figure 3 consists of 50 periods of $\text{InAs}_{0.94}\text{Sb}_{0.06}/\text{In}_{0.98}\text{Ga}_{0.02}$ with layer thicknesses of 85 and 128 Å, respectively. No interface layers were used and a very good match between the observed and calculated pattern was obtained.

The improvement of the x-ray diffraction pattern is due to the minimization of exposure of the InAsSb surface to AsH_3 and/or As. Extended AsH_3/As exposures are known to cause interface roughening in the $\text{InGaAsP}/\text{InP}$ and InAs/GaSb systems [8,9]. Presumably, the presence of AsH_3/As during the purge times in between layer growth is causing the presence of the interface layers. However, the presence of Ga in these layers can not be explained by the alteration in the purging times and gas flows since no TMGa was used during the purges. The presence of TMGa in the interfaces must be due to either the roughness of the surfaces which enables mixing of Ga into the InAsSb layers at the interface or the diffusion of Ga into the InAsSb during growth. In either case, the mechanism for the incorporation of Ga at the interfaces is dependent on the AsH_3/As flux and the time of exposure of the InAsSb surface since the interface was minimized by reducing the purge time and stopping the AsH_3 flow.

Figure 4 - Photoluminescence and lasing spectra for an SLS / InPSb "single heterostructure" up to 100 K. The 95 K and 75 K lasing spectra were obtained at lower spectral resolution than the 14 K lasing spectrum.

A laser SLS structure was grown at atmospheric pressure with a 30 second growth interrupt and AsH₃ flowing during the interrupt. This laser was constructed by growing a 2 μm thick layer of InP_{0.65}Sb_{0.35} on an InAs substrate. A 1.5 μm thick SLS active region was grown on top of the InPSb, and to reduce surface recombination, a 100 $\text{\AA}$ thick InPSb cap layer was grown on top of the SLS. The structure of the active region determined by x-ray analysis was InAs_{0.87}Sb_{0.13} / In_{0.93}Ga_{0.07}As (95 $\text{\AA}$ / 133 $\text{\AA}$ layer thicknesses). The buried InPSb layer and the space above the SLS produced optical confinement within the active region, and optically, the device should operate as a single heterostructure laser.

Laser emission occurred at approximately 320 meV (3.9 μm), and lasing was not observed at ≥ 110 K. In the laser spectra, multiple cavity modes were observed; the closest spacing between modes in the high resolution, 14 K spectrum, was 2.2 meV. Using the index of InAs, 3.52, one obtains a longitudinal mode spacing of 1.74 meV, in rough agreement with the observed value.

The performance of our first SLS midwave laser compares favorably with other prototype lasers constructed in these material systems. An LPE- grown, InAs_{0.95}Sb_{0.05} /

InAs_{0.50}Sb_{0.19}P_{0.31} double heterostructure, injection laser emitting at 3.2 μm had a maximum operating temperature of 110 K [10]. Other double heterostructure lasers that have been demonstrated in this wavelength range include an InAs_{0.91}Sb_{0.09} / Al_{0.5}Ga_{0.5}Sb, optically pumped, 3.9 μm , laser with a maximum operating temperature of 135 K [11], an In_{0.77}Ga_{0.23}As_{0.74}Sb_{0.26} / InP_{0.7}Sb_{0.3} electrically injected laser operating at 3.06 μm and 35 K [1], and an InAs_{0.91}Sb_{0.09} / Al_{0.5}Ga_{0.5}Sb electrically injected laser operating at 3.97 μm up to 80 K [12]. Strain in the active regions of these non-SLS lasers is difficult to determine due to possible errors in alloy compositions and from lattice mismatches (intentional or unintentional). All of these lasers, including our SLS device, probably suffer from a high dislocation density in the active region.

In conclusion, we have demonstrated an improved SLS growth technique and an optically pumped, single heterostructure SLS/InPSb laser which emitted at 3.9 μm with a maximum temperature of approximately 100 K. We anticipate that with improvements in material quality, we may soon realize higher temperature operation of midwave emitters with biaxially compressed InAsSb.

ACKNOWLEDGMENTS

We wish to thank J. A. Bur for providing technical assistance. This work was supported by the US DOE under Contract No. DE-AC04-94AL85000.

REFERENCES

- [1] R. J. Menna, D. R. Capewell, R. U. Martinelli, P. K. York, and R. E. Enstrom, Appl. Phys. Lett. 59, (1991) 2127.
- [2] R. M. Biefeld, J. Crystal Growth 75 (1986) 255.
- [3] R. M. Biefeld, S. R. Kurtz, and S. A. Casalnuovo, J. Crystal Growth 124 (1992) 401.
- [4] R. M. Biefeld, K. C. Baucom, S. R. Kurtz, and D. M. Follstaedt, Mat. Res. Soc. Symp. Proc., Growth, Processing, and Characterization of Semiconductor Heterostructures, Fall 1993, in press.
- [5] S. R. Kurtz and R. M. Biefeld, L. R. Dawson, K. C. Baucom, and A. J. Howard, Appl. Phys. Lett. 64, 812 (1994).
- [6] R. M. Biefeld, C. R. Hills and S. R. Lee, J. Crystal Growth 91 (1988) 515.
- [7] S. R. Kurtz and R. M. Biefeld, Phys. Rev. B 44, 1143 (1991).
- [8] G. Landgren, J. Wallin, and S. Pellegrino, J. Electronic Mater., 21 (1992) 105.
- [9] D. H. Chow, R. H. Miles, J. R. Soderstrom, and T. C. McGill, J. Vac. Sci. Technol. B8 (1990) 710.
- [10] Habib Mani and Andre Joullie, SPIE 1362 (1990) 38.
- [11] J. P. van der Ziel, T. H. Chiu, and W. T. Tsang, Appl. Phys. Lett. 48, (1986) 315.
- [12] S. J. Eglash and H. K. Choi, Appl. Phys. Lett. 64 (1994) 833.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

END

DATE
FILMED

5/16/94

Unc