## Monday Afternoon, September 23, 2019

### MBE

**Room Silver Creek - Session MBE-2MoA** 

**Bismuthides and Antimonides Moderator:** Joshua Zide, University of Delaware

3:30pm MBE-2MoA9 LATE NEWS: Minority Carrier Lifetime and Photoluminescence Properties of Mid-Wave InAsSbBi, Preston T. Webster, Air Force Research Laboratory; P. Petluru, University of Texas at Austin; P.C. Grant, Applied Technology Associates; E.H. Steenbergen, Air Force Research Laboratory; D. Wasserman, University of Texas at Austin

As lower cost infrared imaging goals drive research in alternatives to stateof-the-art HgCdTe, the unparalleled bandgap engineering flexibility afforded by the heaviest group-V element Bi offers a unique III-V analog to HgCdTe in InAsSbBi. By varying the mole fraction of constituent latticematched ternaries  $InAs_{0.91}Sb_{0.09}$  (4  $\mu m$  wavelength at 120 K) and InAs<sub>0.93</sub>Bi<sub>0.07</sub> (12 µm wavelength), guaternary InAsSbBi spans the technologically relevant mid- to long-wave infrared spectrum and can be grown lattice-matched on large area GaSb substrates. Moreover, InAsSbBi's compositional likeness to conventional bulk InAsSb and the InAs/InAsSb superlattice put it in a unique position to take advantage of recent technological innovation discovered and implemented in these related infrared systems. Molecular beam epitaxy grown InAsSbBi alloys are examined using temperature-dependent steady-state and time-resolved photoluminescence spectroscopy, X-ray diffraction, reflection high-energy electron diffraction (RHEED), and Nomarski imaging. RHEED patterns show that the InAsSbBi layer grows with a droplet-free (2×3) surface reconstruction at 380 °C. The surface is observed to remain specular and droplet free during growth, and Nomarski imaging further verifies that a smooth surface morphology is obtained. The InAsSbBi layers are 1 µm thick sandwiched between lattice-matched InAsSb layers providing carrier confinement. Comparison of the tetragonal distortion measured by X-ray diffraction and the bandgap energy measured by steady-state photoluminescence provides an evaluation of the Sb and Bi content of each sample. The target InAsSbBi mole fractions yielding 5 µm wavelength emission are 6.0% Sb and 2.2% Bi, and detailed examination of the photoluminescence properties and molecular beam epitaxy growth conditions show the growth progression towards this goal. Bandgap characteristics and recombination dynamics evaluated from the photoluminescence experiments are compared to equivalent 5 µm wavelength InAs/InAsSb superlattices and 4 µm wavelength latticematched InAsSb bulk samples.

#### 3:45pm MBE-2MoA10 Characterization of Thick GaAsBi Layers Grown with Strain-stabilization, *Margaret Stevens, K. Grossklaus, J. McElearney, S. Lenney, T. Vandervelde*, Tufts University

GaAs<sub>1-x</sub>Bi<sub>x</sub> alloys are challenging to grow by molecular beam epitaxy due to the surfactant-like nature and low solubility of bismuth (Bi) in this system. The key to good Bi incorporation in GaAsBi is to provide enough Bi flux to the surface to stabilize a surfactant layer, but not so much as to induce Ga-Bi droplet formation. The point at which Ga-Bi droplets form, or the Bi saturation point, limits the maximum Bi fraction obtainable with the growth conditions used. Samples grown with Ga-Bi droplets have reduced Bi composition and significant lateral and vertical phase separation. Strainstabilization, by growing on partially relaxed InGaAs buffer layers, can be used to overcome Bi saturation in  $\mathsf{GaAsBi}_{0.07\text{-}0.09}$  films. We propose that reducing the compressive strain decreases the total energy of the system, allowing more Bi to incorporate and Ga-Bi droplet formation to be avoided. We have explored this trend for various GaAsBi epilaver thicknesses, as shown in Figure 1. By growing on In0.105GaAs buffer layers, droplet-free films >100 nm can be achieved for Bi fractions x<0.09. This is an improvement over samples grown with high compressive strain on GaAs, where films of >100 nm are limited to compositions of x<0.07.

In this work, we explore the connections between in-plane strain, Bi incorporation, droplet formation, and maximum GaAsBi film thickness. Samples were grown on a Veeco GENxplor MBE using a valved As<sub>4</sub> source and a solid source effusion cell for group-III elements and Bi. Bismuth fraction was determined by high-resolution x-ray diffraction combined with select samples confirmed through Rutherford Backscattering Spectrometry. Scanning transmission electron microscopy was used to characterize the phase separation brought on by droplet formation. Spectroscopic ellipsometry was used to characterize the absorption coefficient of thick GaAsBi films and identify Urbach tails associated with crystalline disorder.

Lastly, initial doping studies comparing silicon vs. tellurium doping were explored to increase dopant incorporation in this material system. The ultimate goal of this study is to prepare the GaAsBi/InGaAs/GaAs system for optical device applications in the near-IR.

### 4:00pm MBE-2MOA11 Comparing Droplet Formation and Phase Separation in Post-Saturation GaSbBi and GaAsBi, John McElearney, K. Grossklaus, M. Stevens, T. Vandervelde, Tufts University

III-V semiconductors alloyed with dilute amounts of bismuth have been shown to have dramatically reduced bandgaps, making them well suited for optoelectronic applications in the mid- and far-infrared. Given that GaSb has a bandgap already in the near-IR range (Eg = 0.726 eV), GaSb<sub>1</sub>xBix has great potential for pushing out to these long wavelength regimes. Unfortunately, bismuth tends to surface segregate and form droplets at sufficiently high Bi flux rather than incorporate into the growing film. The highest Bi content GaSb<sub>1</sub>xBix reported to date had x=0.14, and only x=0.11 has been achieved without droplets forming on the film surface [1]. We have recently observed that droplets are also likely to form upon reaching some critical thickness for a given Bi content in GaAsBi films, and these droplets can lead to significant lateral and vertical phase separation in the GaAsBi system.

We have found the solubility of Bi in GaSb to be different than the solubility of Bi in GaAs. The same Bi flux that leads to x=0.0315 in GaAs<sub>1×</sub>Bi<sub>x</sub> (grown at 250°C) leads to negligible Bi incorporation in GaSb(Bi) (grown at 285°C). A comparison of the GaSb(Bi) and GaAsBi surfaces is shown in Fig. 1. This work seeks to determine the effect of increasing bismuth flux and film thickness on surface morphology and film homogeneity in GaSbBi films as compared to GaAsBi. Samples were grown on GaSb substrates in a Veeco GENxplor MBE system using a valved Sb cracker and solid source effusion cells for Ga and Bi, with growth monitored in-situ via RHEED. Bismuth content was determined via HRXRD and confirmed for select samples with Rutherford backscatter spectrometry. The presence/absence of droplets was determined via optical microscopy and SEM imaging, while cross-sectional film homogeneity and phase separation was examined via TEM

[1] O. Delorme, L. Cerutti, E. Tournie, J.-B. Rodriguez. J. Cryst. Growth, 447 (2017)

# 4:15pm MBE-2MoA12 Molecular Beam Epitaxy Growth and Bandgap Measurements of InAsSbBi, *Stephen Schaefer*, *R.R. Kosireddy, S. Johnson*, Arizona State University

The molecular beam epitaxy growth of the III-V semiconductor alloy InAsSbBi is investigated for growth temperatures ranging from 400 to 430 °C, As/In flux ratios of 0.91 and 0.94, Sb/In flux ratios of 0.10 and 0.12, and Bi/In flux ratios of 0.05 and 0.10. Bismuth readily incorporates at growth temperatures around 300 °C, but results in material with limited optical quality. Conversely, higher growth temperatures around 400 °C yield improved optical performance, but with limited Bi incorporation. The fraction of the Bi flux incorporated is observed to decay exponentially with a 17 °C characteristic temperature in the high temperature growth regime as shown in Fig. 1. Furthermore, when the As/In flux ratio is increased significantly above stoichiometry, the Bi incorporation decreases as As outcompetes Bi for group V lattice sites as shown by the solid square. Quaternary alloys such as InAsSbBi possess two degrees of freedom that allow the bandgap to be specified independently of strain. Photoluminescence spectroscopy is used to examine the temperature dependent bandgap and optical properties of InAsSbBi, while x-ray diffraction is used to determine strain. The bandgap as a function of temperature is shown in Fig. 2, where an Einstein single oscillator model fit to data (solid curves) provides the zero temperature bandgap energy. A bandgap bowing model is developed and employed to determine the InAsSbBi composition from the measured bandgap and strain.

### 4:30pm MBE-2MoA13 Microstructure, Chemical Composition, and Surface Morphology of InAsSbBi Grown on GaSb by Molecular Beam Epitaxy, *Rajeev Reddy Kosireddy*, S. Schaefer, S. Johnson, Arizona State University

The microstructure, chemical composition, and surface morphology of molecular beam epitaxy grown InAsSbBi is investigated using transmission electron microscopy (TEM), X-ray diffraction, and atomic force microscopy (AFM). The InAsSbBi layers are 210 nm thick and grown at temperatures between 400 and 430 °C on (100) GaSb substrates. The results indicate that the material is nearly lattice matched, coherently strained, and contains dilute Bi mole fractions. The bright field TEM image in Fig. 1 shows no visible defects in the material over large lateral distances. Lateral modulation of the Bi mole fraction is observed in the bright field image and

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in the chemical sensitive 200 dark field image shown in Fig. 2, where a line scan of the image intensity along the black rectangle is inset. Analysis of the ratio of the dark field image intensities indicates that the Bi mole fraction variation has about a 30 nm period and ranges from 0.42% to 0.58% with a 0.50% average value. A rough hazy surface with large Bi-rich droplets on the order of 1  $\mu$ m diameter is observed when the InAsSbBi layer is grown with near stoichiometric As flux (see Fig. 3). Nevertheless, when the As flux is a few percent greater than stoichiometric, a smooth specular surface without large droplets is observed (see Fig. 4). The growth temperature and the As, Sb, and Bi over In flux ratios are listed in each figure. The results at higher growth temperature also show a similar dependence on the As flux. The surface interaction between As and Bi strongly affects the surface morphology and the incorporation of Bi into the InAsSbBi layer.

4:45pm MBE-2MoA14 Dislocation Dynamics as a Function of MBE Growth Conditions in Metamorphic InAsSb, Stephanie Tomasulo, Naval Research Laboratory; C. Affouda, M. Twigg, U.S. Naval Research Laboratory; M. Yakes, Naval Research Laboratory; E. Aifer, U.S. Naval Research Laboratory Long wavelength IR III-V based devices have long been of interest for potential applications such as chemical sensing and large format IR imaging. Within the III-V family, extending to the longest wavelength requires the use of  $InAs_{1-x}Sb_x$  ( $x \le 0.6$ ) which offers the lowest bandgap energy  $(E_g)$  ranging from 0.05-0.35 eV [1]. However, the lack of conventional substrate at the desired lattice constant has restricted progress on the growth and study of this material system. To overcome this limitation, we employ a metamorphic step-graded InAs<sub>1-x</sub>Sb<sub>x</sub> buffer on GaSb, enabling the study of  $low-E_g lnAs_{1-x}Sb_x$  as a function of growth conditions. Using this method, we previously presented the effect of substrate temperature  $(T_{sub})$  and V/III on Sb incorporation of the lowest- $E_g$ cap layer [2]. Here, we investigate the effect of V/III on Sb incorporation as a function of x and use x-ray reciprocal space mapping (RSM) to examine the effect of growth conditions on strain and dislocation dynamics.

We grew several InAs<sub>1-x</sub>Sb<sub>x</sub> step-graded structures in which the Sb/(As+Sb) flux ratio was varied from 0.05 to 0.50 in 0.05 increments, under various T<sub>sub</sub> and V/III, and identified the Sb composition in each layer using RSM along [110] with (004) and (115) reflections. This allows comparison of Sbcontent as a function of Sb/(As+Sb) for various V/III, given in Fig. 1. These results suggest that V/III has little effect on Sb incorporation, in direct conflict with our previous photoluminescence (PL) results [2]. To understand the discrepancy between PL and RSM, we measured (004) RSM of the same three samples with the x-ray beam incident along [1-10], revealing extremely different strain relaxation compared to the [110] case. Asymmetric strain relaxation has been observed in other III-V graded buffer systems and has been explained by different dislocation formation energies and glide velocities along each direction resulting from the core structure of the dislocation being terminated with either a group-III or a group-V element [3]. To develop an understanding of this mechanism within InAs<sub>1-</sub> <sub>x</sub>Sb<sub>x</sub> graded buffers, we will employ the x-ray analysis of Ayers [4] to quantify the threading dislocation density (TDD) in these films along both [110] and [1-10], thus enabling a comparison of TDD as a function of growth conditions, thickness, and propagation direction. AFM and TEM will further support these results. Taken together, we will develop a picture of dislocation dynamics during the growth of metamorphic InAs<sub>1-x</sub>Sb<sub>x</sub>.

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