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1. REPORT DATE (DD-MM-YYYY) 31-01-2023	2. REPORT TYPE Final Report	3. DATES COVERED (From - To) 30-Apr-2020 - 29-Dec-2021
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4. TITLE AND SUBTITLE Final Report: Agile and Repeatable Source for Molecular Beam Epitaxial Growth of Digital and Analog Alloys	5a. CONTRACT NUMBER W911NF-20-1-0087
	5b. GRANT NUMBER
	5c. PROGRAM ELEMENT NUMBER 611103

6. AUTHORS	5d. PROJECT NUMBER
	5e. TASK NUMBER
	5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAMES AND ADDRESSES University of Texas at Austin 101 East 27th Street Suite 5.300 Austin, TX 78712 -1532	8. PERFORMING ORGANIZATION REPORT NUMBER
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9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES) U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211	10. SPONSOR/MONITOR'S ACRONYM(S) ARO
	11. SPONSOR/MONITOR'S REPORT NUMBER(S) 75660-ES-RIP.1

12. DISTRIBUTION AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.
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13. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU	UU		Seth Bank
					19b. TELEPHONE NUMBER 512-471-9669

RPPR Final Report

as of 01-Feb-2023

Agency Code: 21XD

Proposal Number: 75660ESRIP

Agreement Number: W911NF-20-1-0087

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DUNS Number: 170230239

EIN: 746000203

Report Date: 29-Mar-2022

Date Received: 31-Jan-2023

Final Report for Period Beginning 30-Apr-2020 and Ending 29-Dec-2021

Title: Agile and Repeatable Source for Molecular Beam Epitaxial Growth of Digital and Analog Alloys

Begin Performance Period: 30-Apr-2020

End Performance Period: 29-Dec-2021

Report Term: 0-Other

Submitted By: PhD Seth Bank

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Distribution Statement: 1-Approved for public release; distribution is unlimited.

STEM Degrees: 0

STEM Participants:

Major Goals: We enhanced our molecular beam epitaxial (MBE) growth infrastructure for synthesizing emerging photonic materials and devices that operate in the near-, mid-, and far-infrared regions of the electromagnetic spectrum. Specifically, we purchased and installed a Veeco Mark V valved- antimony (corrosive series) cracker source and the associated accessories, which greatly enhanced our materials growth capabilities for mixed arsenide-antimonide-bismide analog and digital alloys.

Accomplishments: This instrumentation has dramatically-enhance our ongoing DoD-sponsored research in several areas, including the synthesis of: (1) AlInAsSb digital alloy materials grown on InP and silicon substrates for near- and mid-infrared low-noise avalanche photodiodes (APD), (2) lattice- matched tunable bandgaps of InAsSbBi alloys grown on InSb substrates, and (3) type-II InAs/InAsSb superlattices on GaSb for power limiters and mid-/far-infrared detectors that exploit emergent light-matter interactions. The Veeco Mark V source features dramatically-improved temporal flux modulation, greatly increased across-wafer flux uniformity, and several other design improvements over previous antimony source. These advantages have significantly improved our ability to fundamentally control the MBE growth process of the aforementioned materials, leading to new avenues in basic materials research, routes to improve discrete device performance, as well as the ability to produce uniform large-area samples necessary to demonstrate focal plane arrays.

These critical new capabilities (lifespan 20-25 years) have dramatically enhanced the PI's – as well as his collaborators' – current, upcoming, and long-term research efforts sponsored by DoD. Indeed, the advances in nanostructured materials that this instrumentation has (and will) enable have manifold application to future strategic DoD capabilities in (multi-modal) sensing, 3- D LIDAR, laser-based countermeasures, free-space communications, security screening, IED detection, and chemical/gas sensing.

It has also provided hands-on experience growing and characterizing new state-of-the-art materials/devices to a number of emerging scientists and engineers. To date, 8 Ph.D. students (6x in PI Bank's group and 2x in Dan Wasserman's group) have directly used the MBE source to advance their research; moreover, 2 Ph.D. students and 3 undergraduate researchers have used materials grown with this source as part of their research.

Please see the attached PDF for more information.

RPPR Final Report

as of 01-Feb-2023

Training Opportunities: The installed instrumentation has provided hands-on experience growing and characterizing new state-of-the-art materials/devices to a number of emerging scientists and engineers. To date, 8 Ph.D. students (6x in PI Bank's group and 2x in collaborator Dan Wasserman's group) have directly used the MBE source to advance their research; moreover, 2 Ph.D. students and 3 undergraduate researchers have used materials grown with this source as part of their research.

Results Dissemination: As this is an instrumentation award, there are no results to disseminate; however, it has already enabled a number of results that are at various stages of publication.

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

Partners

I certify that the information in the report is complete and accurate:

Signature: Seth Robert Bank

Signature Date: 1/31/23 8:13PM

Agile and Repeatable Source for Molecular Beam Epitaxial Growth of Digital and Analog Alloys

Abstract

We enhanced our molecular beam epitaxial (MBE) growth infrastructure for synthesizing emerging photonic materials and devices that operate in the near-, mid-, and far-infrared regions of the electromagnetic spectrum. Specifically, we purchased and installed a Veeco Mark V valved-antimony (corrosive series) cracker source and the associated accessories, which greatly enhanced our materials growth capabilities for mixed arsenide-antimonide-bismide analog and digital alloys.

This instrumentation has dramatically-enhance our ongoing DoD-sponsored research in several areas, including the synthesis of: (1) AlInAsSb digital alloy materials grown on InP and silicon substrates for near- and mid-infrared low-noise avalanche photodiodes (APD), (2) lattice-matched tunable bandgaps of InAsSbBi alloys grown on InSb substrates, and (3) type-II InAs/InAsSb superlattices on GaSb for power limiters and mid-/far-infrared detectors that exploit emergent light-matter interactions. The Veeco Mark V source features dramatically-improved temporal flux modulation, greatly increased across-wafer flux uniformity, and several other design improvements over previous antimony source. These advantages have significantly improved our ability to fundamentally control the MBE growth process of the aforementioned materials, leading to new avenues in basic materials research, routes to improve discrete device performance, as well as the ability to produce uniform large-area samples necessary to demonstrate focal plane arrays.

These critical new capabilities (lifespan 20-25 years) have dramatically enhanced the PI's – as well as his collaborators' – current, upcoming, and long-term research efforts sponsored by DoD. Indeed, the advances in nanostructured materials that this instrumentation has (and will) enable have manifold application to future strategic DoD capabilities in (multi-modal) sensing, 3-D LIDAR, laser-based countermeasures, free-space communications, security screening, IED detection, and chemical/gas sensing. It has also provided hands-on experience growing and characterizing new state-of-the-art materials/devices to a number of emerging scientists and engineers. To date, 8 Ph.D. students (6x in PI Bank's group and 2x in Dan Wasserman's group) have directly used the MBE source to advance their research; moreover, 2 Ph.D. students and 3 undergraduate researchers have used materials grown with this source as part of their research.

1. Overview

The PI is involved in a number of current research efforts sponsored by DoD, with application to strategic capabilities in multi-modal sensing, 3-D LIDAR, laser-based countermeasures, free-space communications, high-speed transistors, security screening, IED detection, chemical/gas sensing, energy harvesting, etc. This work is enabled by the nanoscale design and synthesis of novel materials, including digital and analog¹ alloy semiconductors, using the molecular beam epitaxy (MBE) crystal growth technique.

With this DURIP award, we enhanced our instrumentation for the MBE growth of antimony-containing III-V materials and devices through the acquisition of a Veeco Mark V valved-antimony (corrosive series) cracker source and associated accessories (power supplies and ancillary control/metrology electronics). In **Section 2**, we describe the instrumentation, as well as its acquisition, successful installation, and operation. In **Section 3**, we provide a few illustrative examples how this instrumentation has already enhanced our ongoing DoD-sponsored research in several areas: (1) synthesis of near- and mid-infrared low-noise AlInAsSb digital alloy avalanche photodiode (APD) materials, (2) InAsSbBi alloys for mid-IR and long-wave-IR lattice matched photodetector materials on InSb substrates, and (3) type-II InAs/InAsSb superlattices on GaSb for mid-/far-infrared detectors that exploit emergent light-matter interactions.

This instrumentation represents a strategic DoD investment opportunity to (1) enable future systems based upon new nanostructure-based device technologies and (2) educate graduate and undergraduate students in the strategically important sciences of nanostructured materials, optics, electronics, and optoelectronics.

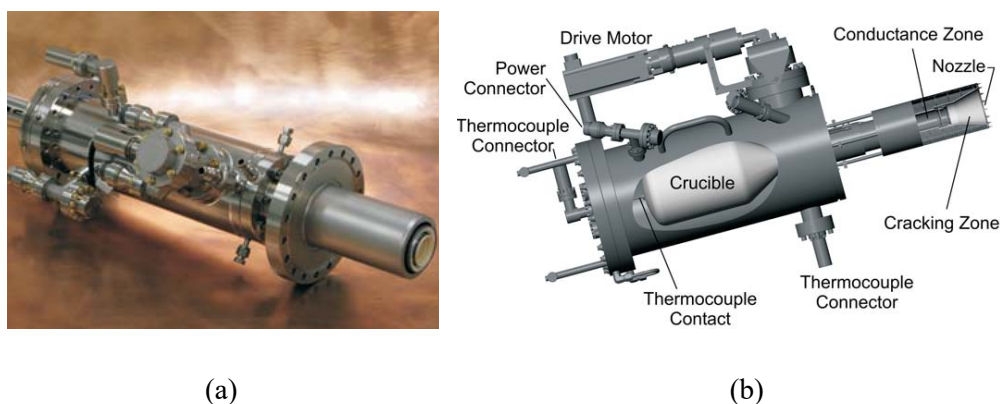


Figure 1. (a) Out-of-vacuum photograph and (b) cutaway drawing of a Veeco Mark V corrosive-series valved cracker. Figures courtesy of Veeco Instruments.

2. Equipment Overview and Installation

The installed instrumentation, shown in **Figure 1**, has greatly enhanced the quality of current research and will enhance the upcoming and future research that the PI is engaged in for the DoD. The equipment was installed at the Microelectronics Research Center of The University

¹ We choose the term ‘analog alloy’ to differentiate conventional alloy semiconductors from digital alloys.

of Texas at Austin, which is an NSF-NNCI supported research center that houses a 12,000 sq-ft cleanroom, with state-of-the-art device fabrication, characterization, and materials growth capabilities. The instrumentation was installed on the PI's As/Sb Varian Gen. II MBE growth system, which is housed in a dedicated class 1000 portion of the cleanroom.

The ordering, receipt, and installation of the instrumentation were all significantly delayed by the COVID-19 pandemic. Placing the order with Veeco was delayed several months due to the logistical issues associated with the remote mode of operation at UT-Austin. In turn, manufacture by Veeco was delayed by an additional several months due to materials shortages and delays with receipt of various subcontracted parts. Installation was then seriously delayed by ~6 more months because the instrumentation arrived while research labs at UT-Austin were operating under serious cohort and social distancing restrictions, which inhibited our ability to safely install the equipment immediately after receipt. The instrumentation is quite heavy, which necessitates several students working in close proximity to physically lift and position the source during installation. Fortunately, the COVID-19 abated sufficiently with the initial vaccine that the cohort and social distancing restrictions were relaxed sufficiently to enable safe installation during an otherwise normal MBE maintenance cycle.

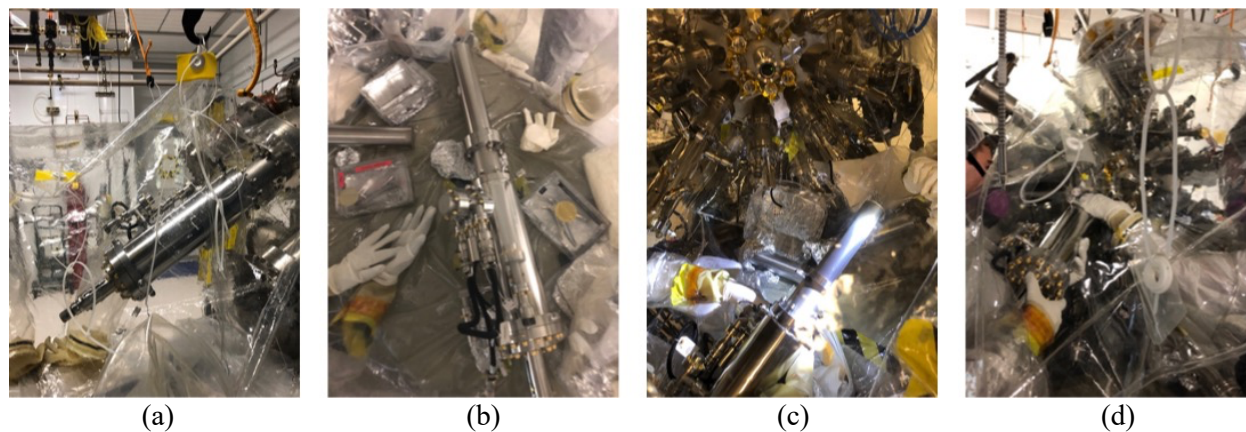


Figure 2. Photographs of the Veeco Mark V valved-antimony cracker at various stages of installation: (a) installed on the test chamber after outgassing, (b) sealed in the glove bag connected to the MBE system, (c) source with blanking assembly removed, and (d) during mounting onto the As-Sb Gen II MBE system.

2.1 Installation and fit-out

The Veeco Mark V valved-antimony cracker source was straightforward to install as it is designed to attach directly to any upward-looking source port on our Varian Gen. II As-Sb MBE system. As shown in **Figure 2a**, we outgassed the source on a separate ultrahigh vacuum test chamber and verified baseline operation of the heaters and valve assembly. We then removed the old antimony source in a nitrogen-purged glove bag that was attached to the MBE system and removed and blanked the new source from the test chamber in a separate glove bag. We then moved the source to the glove bag on the MBE (**Figure 2b**), extensively purged the bag, removed the stainless steel blank (**Figure 2c**), and installed it onto the As/Sb MBE system (**Figure 2d**). We

ran into some electronics issues when our old valve controller failed, but we successfully installed a new Veeco valve controller and were immediately able to control the new source manually with no issues. However, interfacing the new Veeco valve controller with our MBE Control (now part of Riber) Amber growth control software initially proved problematic. We traced this back to a quirk in how Veeco implements RS-485 communications that we were unable to resolve, even after extensive consultation with Veeco; however, we were able to successfully pivot to analog control using a 2404 Eurotherm as an intermediary. Specifically, we communicate valve commands from Amber to the Eurotherm and feed the output from the Eurotherm into analog input of the Veeco valve controller to set the stepper motor (hence valve) position.

Initial tuning of the PID temperature controllers, calibration of the antimony species, and initial flux calibrations (group-III limited reflection high-energy electron diffraction oscillations) were all uneventful for the installed source (**Figure 3**). We have found the flux settling time to be far superior to our old source, consistent with the quoted Veeco specification of <3 seconds. This is important as it was a key driver for requesting this specific instrumentation so that we could experiment with varying the antimony flux within different layer constituents of AlInAsSb digital alloys; these experiments are in progress.

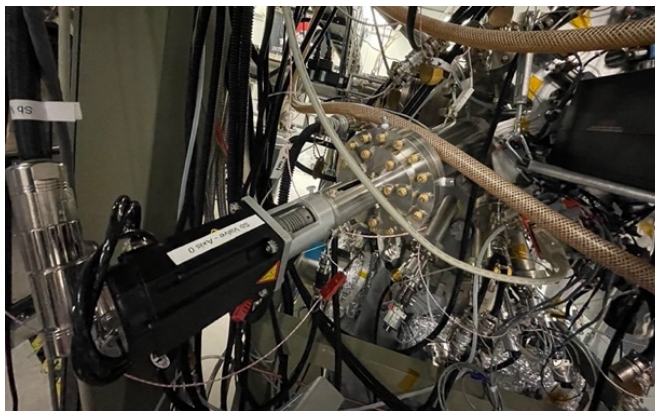


Figure 3. Veeco Mark V valved-antimony cracker installed and operational on the As-Sb Gen II MBE.

The *variability* of the valve hysteresis appears to be significantly improved over the old source; however, the *magnitude* of the valve hysteresis is still higher than the “minimal” value that was quoted. After extensive consultation with Veeco, we believe that the issue may lie with us using a single Eurotherm and dc power supply to heat both the conductance and cracking zones via a series electrical connection. Veeco initially suggested this as a valid operating mode and included a custom connector assembly with the source for implementing series control; however, we plan to implement independent heating of the conductance and cracking zones during our next maintenance cycle, which should remedy this relatively minor issue. We have the necessary additional Eurotherm, dc power supply, and cabling on-hand, but plan to wait to implement it during our next maintenance cycle as (1) it is not an urgent issue and (2) this allows us to avoid unnecessary power cycling of the source.

2.2 Future plans and equipment maintenance

We plan to eventually transfer the old antimony source to an open source port on our B-As-P EPI Gen. II MBE system for new exploratory materials investigations that do not require leading-edge source performance; however, we do not currently have a free source port on this system because of some current experiments. Prior to this we plan to send the old antimony source to Veeco for refurbishment as the valve assembly sticks intermittently and we have been unable to resolve this in-house.

We expect the useful lifetime of the Mark V source to be ~20-25 years, with proper maintenance. This is consistent with the >20-year age of our old Mark IV source, which has broken several times in the past few years, but was fully operational during the PIs first decade at UT-Austin (the source was ~5-8 years old when he joined UT). The Veeco Mark V valved-antimony cracker source is occasionally checked for proper operation and will be calibrated/refilled as-required. We note that, despite heavy usage, we have not yet run out of antimony; this is consistent the predicted ~25% increase in source utilization efficiency that comes from the source nozzle, which is designed to direct the entirety of the antimony flux onto the wafer surface during growth.

3. Exemplar Research Results Enabled by the Instrumentation

This instrumentation has already dramatically-enhance our ongoing DoD-sponsored research in several areas. Here, we describe a few illustrative examples of the research that the Veeco Mark V valved-antimony cracker has enabled/enhanced funded research efforts: (1) AlInAsSb digital alloy materials grown on InP and silicon substrates for near- and mid-infrared low-noise APDs, (2) lattice-matched tunable bandgaps of InAsSbBi alloys grown on InSb substrates, and (3) type-II InAs/InAsSb superlattices on GaSb for mid-/far-infrared focal plane arrays that leverage emergent light-matter interactions.

3.1 AlInAsSb digital alloys grown on alternate substrates

Despite the many possible applications for $\text{Al}_x\text{In}_{1-x}\text{As}_y\text{Sb}_{1-y}$ alloys lattice-matched to GaSb for near- and mid-infrared optoelectronic devices, the growth and optoelectronic properties of this alloy have remained relatively unexplored, due to the presence of a wide miscibility gap. However, the Lester and Dawson groups at University of New Mexico showed that stable AlInAsSb can be grown within the miscibility gap by MBE as a digital alloy of the component binaries, AlAs, AlSb, InAs, and InSb. However, these previous studies were limited to Al fractions ranging from 0% to 40%, as they were more focused on building long-wavelength lasers, and photoluminescence (PL) was only observed up to Al fractions of 30%. We extended this method to cover the entire direct bandgap compositional range of AlInAsSb lattice-matched to GaSb. In collaboration with Prof. Joe Campbell at the University of Virginia, we have been investigating APDs built from continuously graded AlInAsSb digital alloys on GaSb substrates and established it as the first low-noise tunable III-V APD materials system.

With the new Veeco Mark V valved-antimony cracker, we have been able to translate AlInAsSb digital alloy growth to InP substrates. As shown in **Figure 4**, this has enabled us to begin exploring a greater region of the compositional space bounded by the constituent binaries (AlAs, AlSb, InAs, and InSb), beginning with the first growth of lattice-matched digital alloys on InP substrates and InP-on-Si commercial templates.

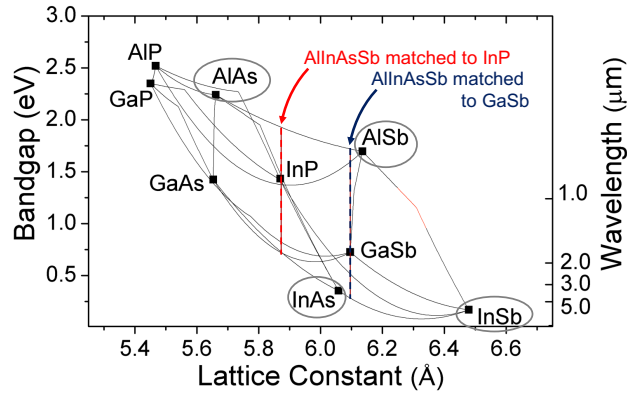


Figure 4. Bandgap versus lattice constant for several traditional III-Vs. Dotted lines indicate lattice-matched bandgap accessible on GaSb (blue) and InP (red).

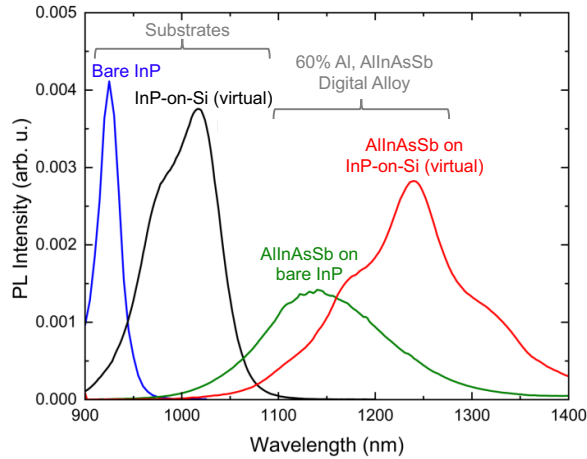


Figure 5. Room temperature photoluminescence of 60% Al average mole fraction AlInAsSb digital alloy films grown on InP and (virtual) InP-on-Si substrates. Spectra for bare InP and InP-on-Si are also shown for comparison.

We began by calibrating lattice-matching to InP with stacked layer structures of different digital alloy layer thicknesses, characterizing them with high-resolution X-ray diffraction (HR-XRD), and performing dynamical simulations to the HR-XRD measurements to extract the constituent layer thicknesses. After lattice-matching, we observed room temperature photoluminescence (PL) from a 300 nm thick AlInAsSb film lattice-matched to InP with 60% average aluminum mole fraction (**Figure 5**) that was capped with 30 nm of $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ to protect against oxidation. In collaboration with Dr. Amy Liu at IQE, we have also been investigating the

growth of these digital alloys on silicon substrates. IQE provided InP-on-Si metamorphic templates that we thermally cleaned and then grew a nominally-identical 60% average aluminum mole fraction AlInAsSb film on the template. We observed the first room-temperature PL from a digital alloy grown on silicon (**Figure 5**), with PL yield being increased and somewhat redshifted compared to the sample grown on bare InP. The lineshape appears to have three distinct spectral regions as well, suggesting further investigations are required; however, these results suggest the tantalizing possibility of future low-noise digital alloy APDs on silicon substrates.

To understand the observed spectral differences, we collected reflection high-energy electron-diffraction (RHEED) images of each substrate before (**Figure 6a** and **6c**) and after (**Figure 6b** and **6d**) digital alloy growth. Qualitatively, RHEED of the growths on InP looked quite similar to what we observed on GaSb; however the RHEED patterns from InP-on-Si were noticeably spottier, both the bare virtual substrate and after digital alloy growth. Notably, the RHEED pattern after digital alloy growth was considerably hazier when grown on InP-on-Si than on bare Si. Consistent with this, atomic force microscopy (AFM) surface scans (**Figure 7**) reveal an increase in root mean square (RMS) roughness after digital alloy growth. Notably, the starting InP-on-Si surface was ~ 2.9 nm RMS roughness, which is significantly higher than the ~ 0.4 nm RMS roughness of our commercial InP substrates. We have received new InP-on-Si virtual substrates from IQE with an improved metamorphic buffer layer scheme that is expected to result in improved digital alloy morphology and plan to grow and characterize photodetectors and avalanche photodiodes built from these materials in the future.

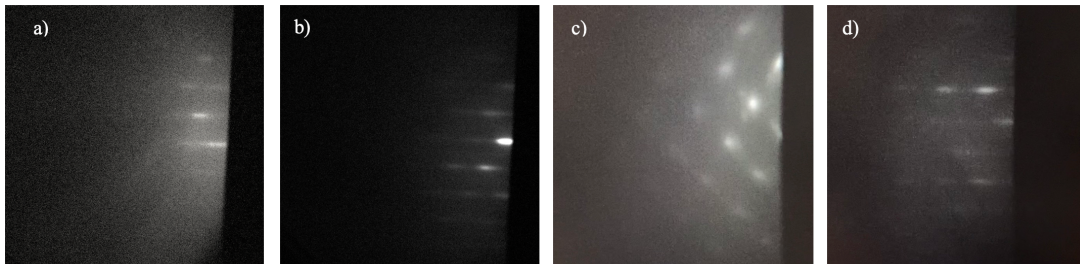


Figure 6. *In situ* reflection high-energy electron-diffractions (RHEED) patterns of (a) bare InP substrate after oxide desorption, (b) during AlInAsSb digital alloy growth on InP, (c) bare InP-on-Si substrate after thermal cleaning, and (d) during AlInAsSb digital alloy grown on InP-on-Si.

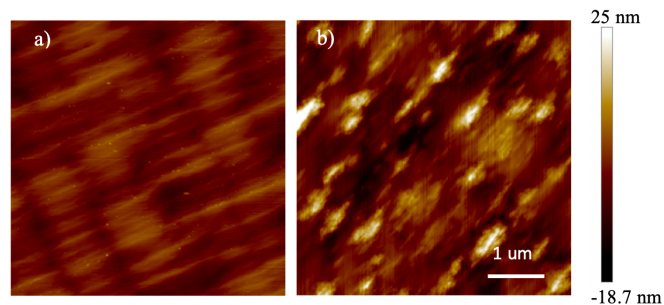


Figure 7. $5 \mu\text{m} \times 5 \mu\text{m}$ atomic force microscopy (AFM) scans of the bare InP-on-Si template (a) before and (b) after AlInAsSb digital alloy growth. RMS roughness increased from ~ 2.9 nm to ~ 5.7 nm, suggesting some additional growth optimization would be beneficial.

3.2 Tunable-bandgap InAsSbBi alloys grown lattice-matched to InSb substrates

As an example of how the improved flux precision of the Veeco Mark V valved-antimony cracker enhances our research, we highlight an example of improved lattice-matching during low-temperature growth near stoichiometry. Bismuth incorporation in III-V alloys induces a desirable bandgap reduction, however, III-V-Bi alloys have struggled to achieve high material quality due to the dramatic difference between the ideal growth temperature of the host III-V matrix and the relatively low growth temperatures necessary for promoting significant bismuth incorporation. InSbBi, however, is a particularly promising material system for accessing the longwave-infrared (LWIR) with high performance optoelectronic devices due to the relatively similar ideal growth windows for InSb and III-Bi materials. Recently we demonstrated the growth of high quality InSbBi with unity sticking Bi incorporation and demonstrated the first photoluminescence (PL) measurements from this alloy. As shown in **Figure 8**, by incorporating As into InAsSbBi, the quaternary can be lattice-matched to InSb substrates enabling the growth of thick layers with tunable bandgaps from ~ 0.26 - 0 eV, which is vital for strong absorption in photodetectors. However, the growth of high-quality InAsSbBi requires extremely tight control of the group-V fluxes to reach stoichiometric fluxes, which are required for high optical-quality.

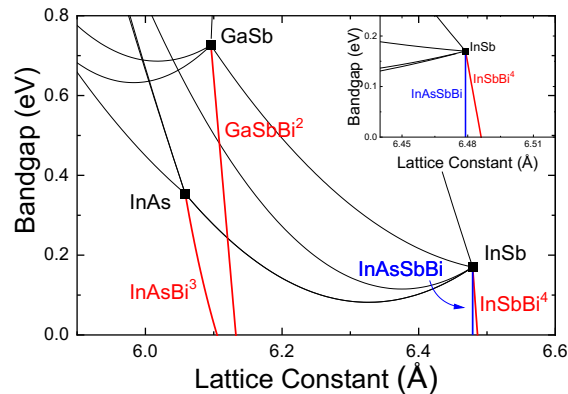


Figure 8. Bandgap energy as a function of lattice constant for $\text{InAs}_{1-x}\text{Bi}_x$, $\text{GaSb}_{1-x}\text{Bi}_x$, and $\text{InSb}_{1-x}\text{Bi}_x$ (red) with inset zoomed in closely around InSb. $\text{InSb}_{1-x}\text{Bi}_x$, in particular, requires relatively little bismuth incorporation to achieve bandgaps down to 0 eV with only small introductions of strain due to the similar size of InSb and InBi. Incorporation of very small concentrations of As enables bandgap engineering opportunities for $\text{InAs}_y\text{Sb}_{1-x-y}\text{Bi}_x$ (blue) lattice-matched to InSb.

InAsSbBi films were grown on (100) InSb substrates. Low substrate temperatures and V/III flux ratios near unity promoted high substitutional bismuth incorporation. Following similar high-quality GaAsBi growth processes, a relatively fast growth rate was used to kinetically suppress bismuth segregation during growth. High-resolution X-ray diffraction ω - 2θ measurements shown in **Figure 9a** of samples grown with different arsenic fluxes showed the expected strain control and eventual lattice-matching with sufficient arsenic flux. Reciprocal space mapping of the lattice-matched film shown in **Figure 9b** confirmed that the film was coherent to the InSb substrate. Rutherford backscattering spectrometry (RBS) measurements (**Figure 10**) were performed by collaborators at Rutgers University to quantify the 1.3% Bi content in the film.

Ion channeling measurements demonstrated highly substitutional bismuth incorporation (~95%), which is a prerequisite for high optical quality.

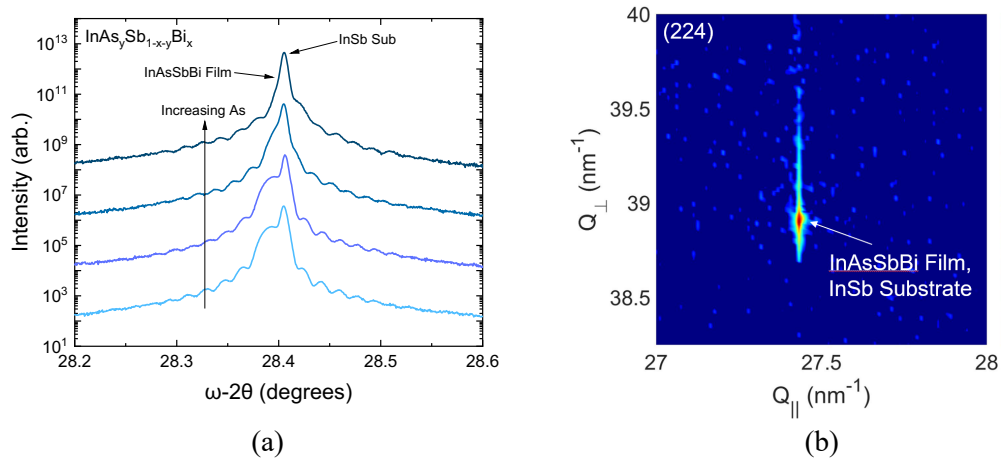


Figure 9. (a) X-ray diffraction ω - 2θ scans of InAsSbBi films about the (004) peak of InSb demonstrate a shift to larger angles with increasing As enabling lattice-matching of InAsSbBi to InSb. (b) A (224) reciprocal space map of InAsSbBi on InSb not only confirms lattice-matching, but also rules out phase separation or relaxation in the film.

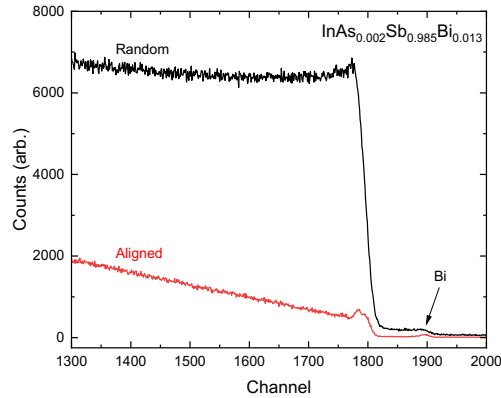


Figure 10. Rutherford backscattering spectrometry (RBS) measurements oriented on both random and channeled crystallographic directions of the sample demonstrating highly substitutional bismuth incorporation in the film of ~95%. Measurements were performed by collaborators at Rutgers University.

As expected from the high substitutional bismuth fraction, we observed room temperature PL at ~7.6 μm from InAsSbBi, as shown in **Figure 11a**, which is significantly redshifted from the emission peak of InSb. As shown in **Figure 11b**, peak emission redshifted with increasing temperature, exhibiting Varshni parameters consistent with a band-to-band optical transition. Experiments are planned to demonstrate InAsSbBi metal-semiconductor-metal photodetectors, as well as doping experiments to realize pin- and nBn-type detectors as well.

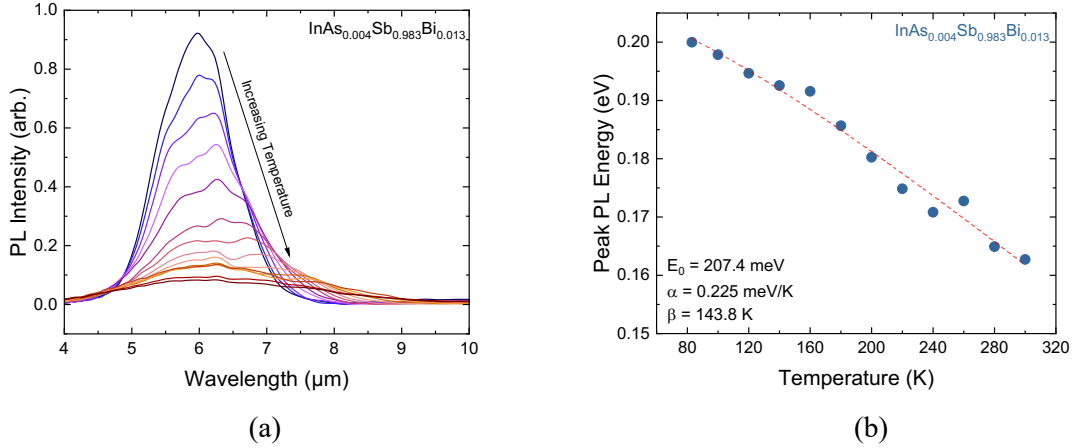


Figure 11. (a) Temperature-dependent photoluminescence measurements of lattice-matched InAsSbBi demonstrate decreasing energy with increasing temperature consistent with an interband transition. (b) A Varshni fit to the PL data yields reasonable Varshni parameters (α and β) consistent with a band-to-band optical transition.

3.3 Type-II superlattice focal plane arrays harnessing emergent light-matter interactions

To highlight how the increased flux uniformity of the Veeco Mark V valved-antimony cracker has enhanced our research, we present some work by Prof. Dan Wasserman’s group who uses our MBE systems to grow advanced type-II InAs/InAsSb superlattice detectors. In all of our prior GaSb-based device experiments, we had employed 2” substrates because of poor flux uniformity across larger diameter wafers. However, this is problematic for transitioning research from discrete devices to large area focal plane arrays because of the prohibitively small wafer area for refining the array fabrication process. The new source has enabled us to transition growths for detector arrays from 2” to 3” substrates, which will accelerate our focal plane array work.

These detectors leverage guided mode resonance (GMR) structures to enhance absorption and minimize dark current in ultrathin, all-epitaxial mid-wave infrared detectors that operate at room-temperature. As shown in **Figure 12a** and **12b**, resonance occurs by coupling to a mode weakly confined to a high-index dielectric grating layer that is sandwiched between two low-index layers such that light from this leaky grating mode can interfere with reflected or transmitted light. This all-epitaxial approach to GMR enhancement leverages the high refractive index of GaSb and the low-index of highly doped (n^{++}) semiconductor materials to enable coupling to a GMR mode within the absorbing region of an nBn detector (**Figure 12c**). The resulting improvement in absorption (**Figure 12d**) allows the detector to use an extremely subwavelength absorbing region to minimize dark current in the device without sacrificing external quantum efficiency, including when operated with substrate side illumination for integration into a focal plane array. Thanks to the improved flux uniformity of the new antimony source, their most recent iteration of this device structure (**Figure 13a**) was able to be grown on a 3” GaSb substrate with high structural quality as seen in HR-XRD (**Figure 13b**) to offer compatibility with existing fabrication processes for the

GMR detector and a readout integrated circuit. Large area focal plane arrays are currently under fabrication by their commercial partner.

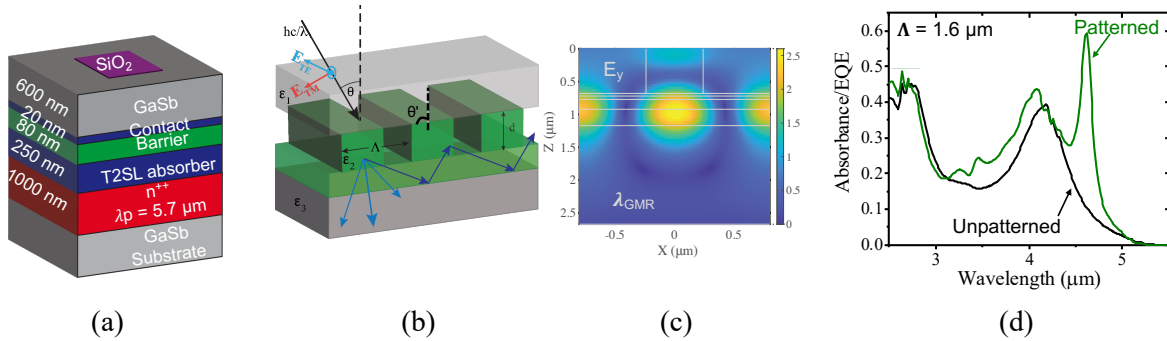


Figure 12. Guided-mode resonance detector architecture. (a) Layer structure using a SiO_2 surface grating and a n^{++} InAs plasmonic layer to enhance (b) the electric field profile overlapping with the T2SL absorber to (d) enhance resonant absorption and detector external quantum efficiency (EQE).

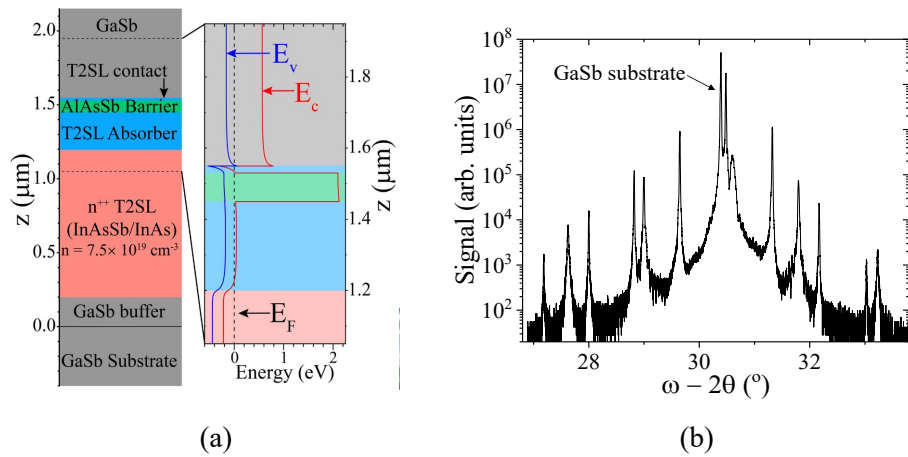


Figure 13. (a) Guided-mode resonance nBn device layer structure and corresponding band diagram for initial focal plane array experiments. (b) X-ray diffraction scan of the structure grown on a 3" GaSb substrate for focal plane array demonstration. The pronounced superlattice fringes suggest excellent structural quality.