R2-B.4: Mid-Infrared Photonic Integrated Circuits for Stand-Off Detection of Trace Explosives

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II. PROJECT DESCRIPTION

A. *Project Overview*

This project aims to develop a mid-infrared photonic integrated circuit (MIR-PIC) and use the device for stand-off detection of trace explosives in the solid phase. The proposed MIR-PIC is a mid-infrared (mid-IR) heterodyne receiver where a Schottky barrier diode has been integrated into a high-performance, mid-IR quantum cascade laser (QCL). The final year of this project focuses on leveraging highly scalable semiconductor growth and metal-organic chemical vapor deposition (MOCVD), to demonstrate high-performance devices that can be transitioned to industry. We also explored alternative device designs, to simplify fabrication, including all-optical lithography and a dual-use top contact that provides current injection and a nonlinear diode response.

This research addresses the void of high-performance, compact technologies capable of measuring the phase and amplitude of mid-IR light that has interacted with a sample under test. This semiconductor transceiver operates by mixing light scattered off the sample under test and coupled back into the QCL waveguide with the internal field of the waveguide. Changes in the phase and amplitude of the scattered light are detected by measuring the voltage over the integrated diode.

Compared to existing optical stand-off detection technologies, there is no need for an external detector or optics, as the entire sensor operates at room temperature, and the sensitivity and detection limits are anticipated to improve by orders of magnitude. The proposed MIR-PIC is ultracompact (~5 mm × 300 mm) and low cost, making it appropriate for commercial-scale production, and it can be integrated into large format arrays for imaging. Single devices will enable rapid stand-off detection of explosives, and arrays of these devices will enable imaging with phase, amplitude, and spectral content for improved detection.

The MIR-PIC represents a fundamentally new type of mid-IR semiconductor transceiver that will enable phase- and amplitude-sensitive imaging in the mid-IR via an ultracompact device. This research will have significant impact on the homeland security enterprise due to the complementary sensing and imaging modalities the MIR-PICs enable, as well as the low-cost, small footprint, and improved sensitivity of these devices.

Ultimately, the sensors can be used for detecting explosives residues on skin, clothing, personal items (travel bags, briefcases, etc.), containers, vehicles, and other substrates. Myriad other fields—including medicine, drug enforcement, and environmental monitoring—will also benefit.

B. State of the Art and Technical Approach

B.1. State of the Art Synopsis

Mid-infrared light interacts strongly with the fundamental vibrational and rotational modes of molecules. This strong light-matter interaction results in molecular fingerprints across the mid-IR that can be used for sensitive detection of trace amounts of molecules of interest [1, 2]. The mid-IR is playing an increasing role in the detection of trace gasses and materials across many fields, including the detection of explosives and their precursors. This State of the Art Synopsis focuses on stand-off detection using mid-infrared light and the mid-IR optical sources used in these measurements.

B.2. Mid-Infrared Stand-Off Detection

Passive infrared stand-off detection of explosives measures the spectrally dependent emissivity of a sample under test. Typically, the thermal emission spectrum is measured using a Fourier transform infrared spectrometer (FTIR) and a cooled HgCdTe detector. Hyperspectral imaging using a mid-IR focal plane array and FTIR have been used to detect trace amounts of explosives [3, 4]. However, the use of passive stand-off detection in real-world scenarios is ultimately limited by the low thermal emission of the sample (sub-μW), the cost and size of the FTIR and detector, and the requirement of cryogenically cooled detectors.

Active stand-off detection uses an optical source to illuminate the sample under test. Here, we focus on coherent illumination since the delivered power is much higher and the beam can be collimated. Tunable CO_2 lasers and optical parametric oscillators have been used as sources previously [5, 6], but these systems are prohibitively expensive, large, and cumbersome for wide-scale deployment. Mid-infrared QCLs are rapidly becoming the source of choice for active stand-off detection because they are commercially available over much of the mid-IR spectrum, emit watt-level power, operate at room temperature, are spectrally tunable via temperature and/or voltage, and are compact [7]. Active stand-off detection of trace explosives is typically achieved by measuring the diffuse reflection of light scattered from an illuminated sample, and the measured spectrum is compared to a library of spectra for explosives [8-10]. Recent progress using QCLs as the optical source has focused on narrow line-width lasers for gas sensing [11], improved sensitivity [12], and increased spectral coverage [13].

For most state-of-the-art sensors, the sensitivity of the system is limited by the relative intensity noise (RIN) of the QCL because a low-noise detector and amplified chain are employed [14]. While the low-noise detector and amplifiers are sufficient for laboratory work, they are not desirable for wide-scale deployment due to the added size, weight, cost, and complexity. The RIN of lasers has been suppressed using many techniques including balanced detectors, active laser intensity stabilization, and heterodyne detection [14]. Mid-IR balanced detectors are difficult to obtain and must be cryogenically cooled, and intensity stabilization adds significant complexity and cost to the spectroscopy setup. Heterodyne detection has been achieved using discrete optical components. Heterodyne-enhanced detection of NO has been demonstrated using a mid-IR

QCL and a room-temperature HgCdTe detector; the overall sensor operates at 3.7 times the fundamental quantum shot limit [14]. Similar heterodyne techniques have been applied to explosives detection in the mid-IR, but the optical configuration of the sensor is not practical for field work—our project integrates the source and mixer into a single device.

Terahertz (THz) QCLs with integrated Schottky barrier diodes for heterodyne detection have been demonstrated by a member of our team (Wanke) [15, 16]. These transceivers operated at cryogenic temperatures due to challenges with the THz QCL active core, and performance was limited in part by the internal field of the THz QCL. A challenge with realizing heterodyne detection in a mid-IR QCL is the need for a high-performance laser with large internal fields.

B.3. High-Performance Mid-Infrared Quantum Cascade Lasers

Mid-IR QCLs were first demonstrated twenty-two years ago [17]. Since then, the performance of QCLs has rapidly and drastically increased to include room-temperature continuous-wave operation, single-mode emission, multi-wavelength and tunable broadband lasing, high wall-plug efficiency (WPE) operation, and more [7]. QCLs are now available from several commercial vendors, and Thorlabs has some lasers available for same-day shipping [18]. Recent progress on QCLs has focused on improving the output power and WPE (electrical to optical conversion efficiency) of the devices [19, 20], broadening the gain spectrum [21], and generating frequency combs [22]. Much of the progress in high-power, high-WPE QCLs has been enabled by carefully considering the effects of interface roughness on the optical and electrical properties of the lasers [20, 23–26]; this work will be key to the MIR-PICs in this project and Hoffman is involved in this area of research.

B.4. Technical Approach

Figure 1 is a schematic of a MIR-PIC deployed as a sensor. The device comprises a mid-IR waveguide (blue, horizontal line) with a mid-IR QCL active core (the active core resides inside of the waveguide), and an integrated Schottky barrier diode (gold dot).

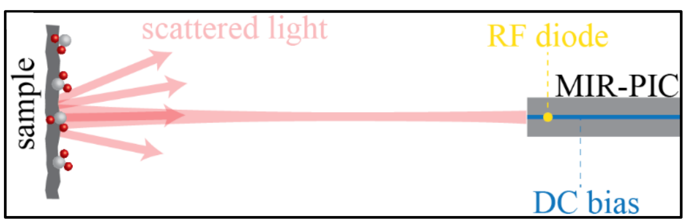

Figure 1: Schematic for stand-off detection of solid phase trace explosives using a MIR-PIC.

A schematic of a MIR-PIC is depicted in Figure 2. The active core of the QCL generates mid-IR photons via electron transitions between the bound sub-bands of hundreds of coupled quantum wells which are then guided in the waveguide. During operation, some of the generated photons are emitted from the facet of the waveguide, scatter off of the sample under test (*c.a.* 3" to 12" away), and re-enter the waveguide. These photons are then mixed by the Schottky diode with the internal field (photons) of the waveguide, Figure 3. This nonlinear mixing results in a voltage over the diode that oscillates at the difference frequency between the internal field and the light that re-entered the cavity, called the intermediate frequency (IF). For a multi-mode Fabry-Perot cavity, a mixing response in the diode is observed at the free spectral range (mode spacing) [15, 16]. For cavities ~3 mm long, the IF is ~14 GHz, a frequency that is easily accessible using common microwave equipment available in our laboratories.

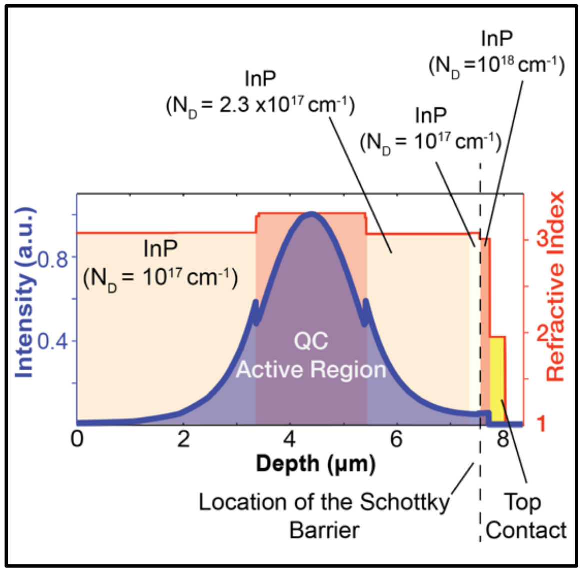

Figure 2: New InP-based design. This design is being grown by Adtech Optics via MOCVD. The mode profile and the refractive index of the device is shown. The mode intensity at the Schottky Diode is designed to be 3% of its maximum intensity.

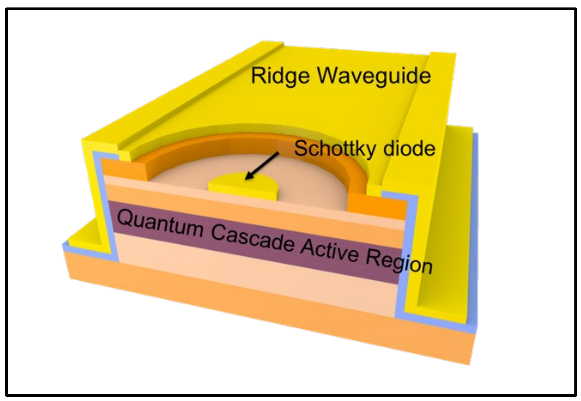

Figure 3: Schematic of a MIR-PIC, highlighting the high-performance quantum cascade active region design, Schottky barrier diode, and ridge waveguide.

While mixing has been demonstrated in THz photonic integrated circuits [24, 25], our project aims to develop a platform for the mid-infrared. This is an important advancement for two reasons. First, mid-infrared quantum cascade active regions have been demonstrated to operate at room temperature in continuous-wave mode. Such performance is extremely important for commercial transition of these devices. Second, mid-infrared light interacts strongly with fundamental vibrational and rotational modes of molecules. This strong light-matter interaction ultimately directly influences the detection limits of the sensors.

The final year of this project focused on advancing the MIR-PIC technology to lower the barrier for commercial transition. We investigated two pathways to achieve this goal. The first approach is to demonstrate room-temperature operation of a MIR-PIC operating in CW mode for devices fabricated from wafers grown via metal organic chemical vapor deposition (MOCVD). Our second approach is to demonstrate nonlinear mixing on the top contact of the waveguide ridge, rather than on a Schottky barrier diode. This will reduce the complexity of the fabrication process, reducing the cost per device and potentially improving device yield and performance.

To realize these Year 7 goals, we developed collaborations with Sandia National Laboratories (SNL) and Adtech Photonics, Inc. (Adtech). Dr. John Klem at

SNL, through a Center for Integrated Nanotechnologies User Proposal, performed strain-balanced growth of two wafers via molecular beam epitaxy (MBE) for MIR-PICs that incorporate and forgo the Schottky diode. Adtech provided heavily subsidized growth of MOCVD-grown wafers.

Figure 4 shows a portion of the conduction band that we have designed for strain-balanced growth on InP substrates via MBE. The calculated figure of merit for the design is comparable to some of the highest performance devices designed to date. Figure 4 depicts the waveguide design that accompanies this active region design. While the calculated optical loss of the waveguide is slightly higher than the highest performing devices, it is well within the range of high-performance waveguide designs.

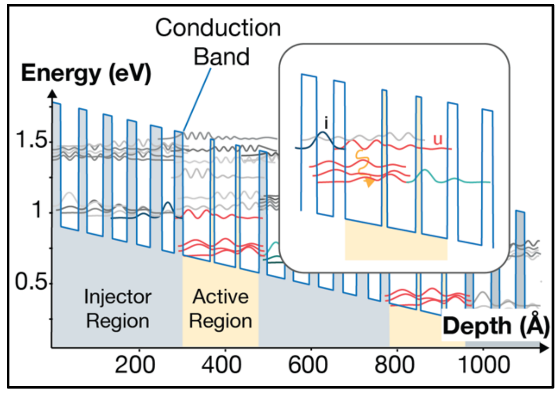

Figure 4: New active region design. Two periods of the conduction band of the active region for strain-balanced In0.66GaAs/ In0.31AlAs on InP. The grey and red curves are the calculated single electron wavefunctions. The curves in red and green are most closely related to the designed optical transition. The applied field is 73.1 kV/cm, and the optical transition is at 6.29 µm.

A distinction between these new devices and our previously tested designs is that the waveguide incorporates a thick layer of InP. For QCLs, an InP waveguide is desirable due to the superior thermal conductivity of the material, which allows efficient extraction of heat from the active region. For a MIR-PIC, the temperature performance is similarly expected to improve, but it is not clear how the overall performance of the MIR-PIC will be influenced. The main reason for this uncertainty is the higher ideality factor for diodes fabricated in InP. Our waveguide design incorporates strategies to mitigate the higher ideality factor. Growth of this heterostructure was completed by Adtech on May 2, 2019.

We fabricated structures for understanding electron transport in these devices and laser ridges to understand the optical properties (e.g., optical gain) of the design structure. An example of the optical gain spectrum measured for a characteristic device is shown in Figure 5. We measured the gain coefficient and the waveguide loss of the devices. In general, the gain coefficient is large (12.09 cm^{-1}/J), but it is still smaller than the highest performing device. We do notice larger waveguide loss in the devices that try to leverage the top metal as the nonlinear diode (16 cm^{-1}); the loss is about 50% greater than conventional QCLs. Gain measurements, along with parameters such as the turn-on voltage, differential resistance, and operating voltage—all of which can be temperature-dependent—can be related to the design and the performance of lasers and MIR-PICs.

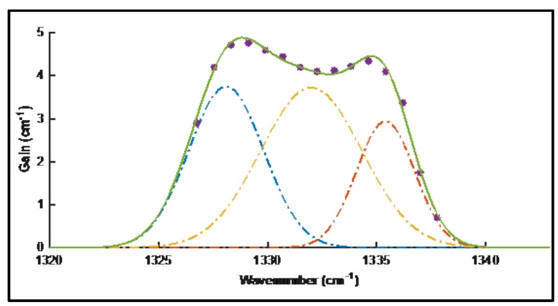

Figure 5: Gain spectrum obtained from Hakki-Paoli calculations. Experimental data (purple stars) was acquired at 80K, and a Gaussian fit was used to estimate the gain shape (green line). Individual transitions that make up the gain shape are depicted in dashed lines

Our next step in the characterization is to measure the microwave response of the MIR-PIC by measuring the voltage over the diode. Figure 6 is a schematic of two circuits we have implemented for testing devices (a) with integrated diodes and (b) without integrated diodes. During this characterization, the devices are operated in continuous wave mode (constant voltage/current). A bias-T is used to couple only the microwave signal into a network analyzer for measuring the spectrum of the generated microwave signal.

The full characterization of the devices was impeded by laboratory closures due to COVID-19. Figure 7 depicts the microwave difference frequency of the amplified spontaneous emission (ASE) spectrum. Our equipment is capable of measuring AC signals below 35 GHz, and there is a strong spectral response around 25 GHz. While the laboratories were shut down, we developed equivalent circuit models and numerical models for MIR-PICs operating at 25 GHz. While we did not originally intend to develop these models as part of this program, they have provided valuable information on improving the overall MIR-PIC performance.

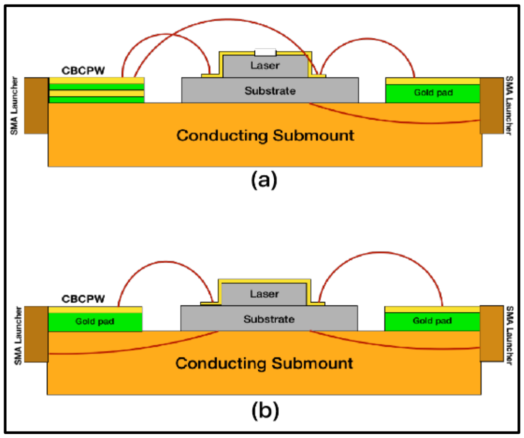

Figure 6: Wiring schematics mixing experiments. (a) Wiring diagram for laser with the diode on. CBCPW is isolated from the ground of the laser. (b) Wiring diagram for laser without a diode. Contacts also used as the nonlinear element. Ground of CBCPW is connected to the laser ground.

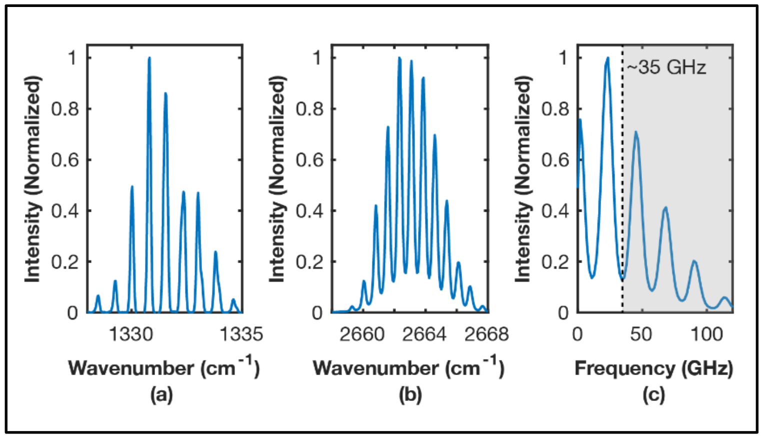

Figure 7: ASE spectrum and calculated sum/difference frequency generation. (a) ASE spectrum acquired at 80K (experimental data). (b) Simulated sum frequency generation. (c) Simulated difference frequency generation. Only signals below ~35 GHz can be detected because of equipment limitations.

An equivalent circuit model for a MIR-PIC and all associated circuitry is given in Figure 8. One immediate insight from these models that we gained is the short stub with designed impedance Z_1 that is now incorporated into our device design, Figure 8.

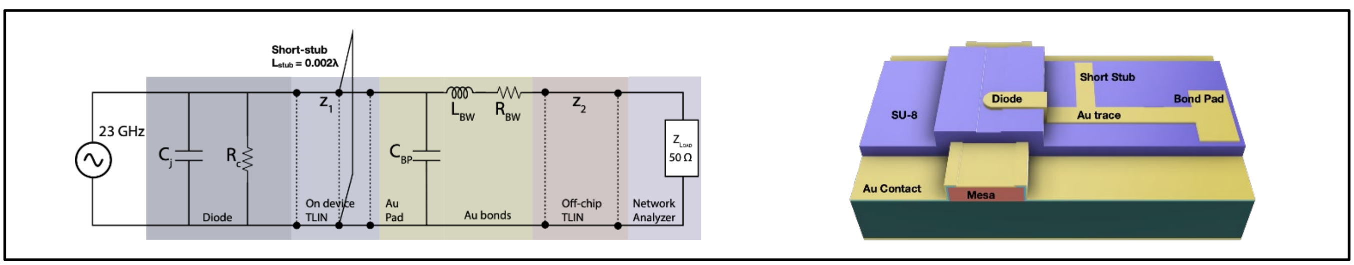

Figure 8: Equivalent circuit model for microwave feed line at 23 GHz and updated device design. (left) 23 GHz corresponds to the difference frequency generated by the Schottky Diode. Impedance matching is done off-chip to increase the flexibility of the design. (right) SU-8 is used as a dielectric layer because of its low price, low loss tangent, and low dielectric coefficient at 24 GHz.

In addition to the equivalent circuit models, we developed models in a standard commercial microwave package to better analyze design devices and circuits. Smith charts depicting the input impedance, Z_{in}, at 48 THz (optical frequencies) and 10 to 30 GHz (microwave frequencies) are given in Figure 9. These models are useful for impedance matching circuits to the MIR-PIC and are available by contacting PI Hoffman.

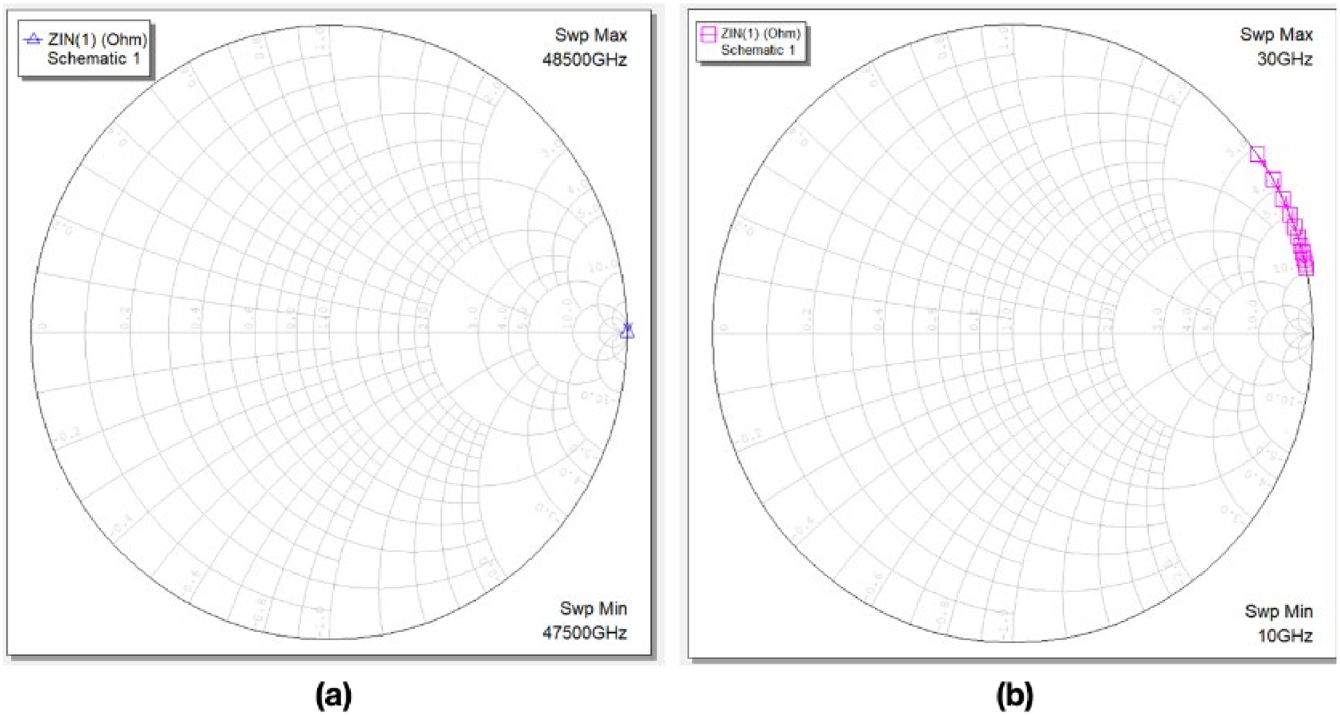

Figure 9: Smith charts showing the input impedance of the MIR-PIC (a) at frequencies from 47.5 THz to 48.5 THz. (b) at frequencies from 10 GHz to 30 GHz.

C. Major Contributions

Year 7:

- Designed a quantum active region to compare MOCVD- and MBE-grown MIR-PICs. The transceivers had differing waveguides, as necessitated by the available materials.
- Fabricated and characterized MIR-PIC devices for semiconductor wafers grown via MBE and MOCVD.
- Developed rigorous microwave models for the entire MIR-PIC device using the industry-standard, commercial software package Microwave Studio. These models are available by contacting Hoffman.
- Developed equivalent circuit models for the MIR-PIC transceiver that are useful for studying trade-offs in device performance and circuit design.
- Developed alternate MIR-PIC fabrication strategies to reduce fabrication costs and improve device yield. These new approaches reduce the complexity of fabrication. We have pursued multiple improvements: replacing air bridges with contacts deposited on benzocyclobutene (BCB); fabricating the diodes using photolithography; and, fabricating devices with the entire top contact serving as the Schottky diode.
- Accessed MIR-PICs that use the top metal as the mixing diode and device contact.
- Developed coplanar waveguide submounts with impedance matching to efficiently extract the microwave signal from the MIR-PIC.

Year 6:

- We made significant updates to our simulation and design software to enable designs in InP-based material systems. This was needed because we shifted our focus to InP-based devices to demonstrate the feasibility of transition for these devices.
- We fabricated devices from a new wafer grown by Adtech (using MOCVD) and devices from wafers grown by Sandia National Laboratories (using MBE).
- We have improved the intermediate frequency amplitude by approximately four times.

Year 5:

- We developed numerical code to model the quantum and optical properties of the MIR-PICs. Using these models, we designed MIR-PIC devices to address problems with the devices used for our preliminary study that prevented the devices from operating in continuous wave mode. Specifically, we developed a quantum cascade gain region capable of providing double the gain of our previous design. These new active region designs employ ultrastrong coupling to improve the injection of electrons into the upper-laser level.
- We used our optical models to design new optical waveguides, focusing on the optical loss, field strength at the metal contact, and confinement factor. We reduced the optical loss by approximately 60% by modifying the field strength at the metal contact and the doping of the cladding layers. The field strength at the metal contact is now 20% of the peak field in the active region; we estimate that this will be sufficient for the MIR-PICs in this project. In order to improve gain, we have also increased the optical confinement factor by ~30% by adding additional active regions. We expect that these changes should enable continuous wave operation of the MIR-PIC.
- We fabricated MIR-PIC devices and performed basic electrical and optical characterization. The devices operated in pulsed and continuous wave mode.

D. Milestones

- Compare the performance of MIR-PICs grown via MBE and MOCVD.
    - Wafers grown via MBE and MOCVD have both been processed, and characterization is complete on the MOCVD devices. Delays due to equipment failure in the Notre Dame Nanofabrication Facility and COVID-19 have slowed testing of the MBE-grown samples.
- Design, fabricate, and characterize a MIR-PIC that allows mixing on the top device contact rather than a separate Schottky diode.
    - Devices have been designed and fabricated. Continuous-wave characterization has been performed, but laboratory closures due to COVID-19 have prevented full microwave characterization.
- Determine the detection sensitivity of devices without an integrated diode for trace solid phase explosives.
    - While this research is still planned for June, the laboratory closure has significantly delayed the experimental characterization. However, during the closure, we have developed rigorous microwave models that could be used for estimating this limit or better optimizing devices for sensitivity.
- Develop rigorous microwave models for MIR-PICs.
    - We designed microwave models for the entire MIR-PIC package that incorporate realistic device parameters. In many cases the device parameters are from actual experiments. These models can be used to analyze trade-offs between the package design and the MIR-PIC performance.

There were three delays that slowed progress in Year 7:

1. MBE semiconductor growth
2. cleanroom down time
3. laboratory closures due to COVID-19

Our mitigation strategies for each are as follows.

1. While we were waiting on the MBE wafers, we focused our fabrication and characterization efforts on the MOCVD samples. (60%)
2. Our fabrication flow required reactive ion etching; however, the cleanroom equipment required for the etch was unavailable from January to March due to a number of cascading failures. We therefore developed wet-etch recipes for the fabrication of the MIR-PICs. (100%)
3. During the laboratory closure, we focused on developing microwave models for the MIR-PICs. (100%)

E. Final Results at Project Completion (Year 7)

- Demonstrated novel, ultracompact MIR-PIC transceivers that operate as both a source and receiver.
- Compared MIR-PICs fabricated using MBE- and MOCVD-grown wafers to investigate commercially scalable technologies
- Developed rigorous microwave and quantum models for MIR-PIC design.
- Engaged leading companies in mid-infrared devices (Thorlabs and Adtech Photonics) for transitioning MIR-PICs to a commercial product.

III. RELEVANCE AND TRANSITION

A. Relevance of Research to the DHS Enterprise

This project addresses the need for ultracompact, sensitive sensors for the stand-off detection of trace explosives in the solid phase (ng/cm^2). Such capabilities could be integrated into handheld devices, unmanned aerial vehicles, remote operated vehicles, and existing security-screening infrastructure as primary or confirming sensors.

- Stand-off detection of explosives is critical for the safety of Homeland Security personnel. We will demonstrate stand-off detection at distances greater than 3 feet but not likely until spring of 2021 at the earliest.
- Compact sensors with high detection sensitivity are needed for the detection of trace explosives. We will demonstrate detection of an explosive in the solid phase. Our sensitivity targets are sub-ng/cm^2; however, since this sensor is entirely new, these detection limits may not be achievable immediately or within the time frame of this project. A goal of this project is to study the relationship between the design and performance of the MIR-PIC and the detection limits when the MIR-PIC is used as a sensor.

Sensitive, stand-off detection could have a transformative impact on the detection of explosives by enabling widespread screening of individuals, vehicles, and objects. The high sensitivity could enable the use of these devices as a primary or confirming sensor, and the ultracompact footprint could enable handheld deployment.

B. Status of Transition at Project End

Our vision for this technology within the homeland security enterprise is to develop a linear array of MIR-PICs for rapidly scanning and imaging letters and packages in sorting facilities or baggage and personal items in screening stations at airports. The array of MIR-PICs would comprise devices that target different spectral regions, enabling preliminary detection of many different solid phase trace explosives or contaminants. We have developed single MIR-PICs and are now working toward scaling those devices into arrays. We are now collaborating with Adtech Photonics, Inc., a leader in QCL technologies. Adtech Photonics employs state-of-the-art growth using MOCVD, a highly scalable semiconductor growth technology. Professor Hoffman has collaborated with several members of the technical staff, and together they have demonstrated world-record QCLs. Mary Fong, the CEO of Adtech Photonics, has directly expressed interest in commercializing the technology in this project.

C. Customer Connections

- Mary Fong, Adtech Photonics LLC. Frequency of contact varied depending on the project status. Adtech Photonics provided semiconductor growth via MOCVD for this project at a heavily-subsidized cost. We have had conversations regarding transitioning to a product.
- Yamac Dikmelik, Thorlabs. Frequency of contact was less than quarterly. We have had discussions with Thorlabs about transitioning the MIR-PICs to a product. Those conversations guided the Year 7 goals.

IV. PROJECT ACCOMPLISHMENTS AND DOCUMENTATION

A. Education and Workforce Development Activities

1. Course, Seminar, and/or Workshop Development
   a. Hoffman incorporates aspects of this research into his graduate-level Nanophotonics and Quantum Optics course. He is also developing an Advanced Condensed Matter course that will include lectures discussing topics relevant to the design of semiconductor devices for optical detection of explosives.
2. Student Internship, Job, and/or Research Opportunities
   a. Undergraduate students working in Hoffman's laboratory participate in group meetings where they are engaged in the research in this project and the needs of DHS.
3. Interactions and Outreach to K–12, Community College, and/or Minority-Serving Institution Students or Faculty
   a. Hoffman has participated in the Indiana STARBASE Program, a DOD-sponsored program for introducing K–12 students to STEM in "hands-on" activities.

B. Peer Reviewed Journal Articles

Pending –

1. Harden, G., Lee, K., Jena, D., Xing, H., & Hoffman, A.J. "Optical Gain and Gain Saturation in Optically-Pumped AlGaN/AlN Quantum Wells." In preparation.

C. Student Theses or Dissertations Produced from This Project

1. Ahmet Cagri Aydinkarahaliloglu will defend his PhD candidacy in fall 2020 with the planned title, "Mid-Infrared Photonic Integrated Transceivers for Intermode Heterodyne Mixing."

D. New and Existing Courses Developed and Student Enrollment

New or Existing	Course/Module/ Degree/Cert.	Title	Description	Student Enrollment
Existing	Course	Nanophotonics and Quantum Optics	This graduate-level course discusses the fundamentals of nanophotonics, including mid-infrared light-matter interactions and quantum well semiconductor devices, which are central to this project	15

E. Software Developed

1. Models
    a. We developed microwave models in industry-standard software packages for the entire MIR-PIC package. Currently, these are available by contacting the PI. We are happy to provide them through an alternative mechanism though.
2. Other
    a. We developed software for controlling mid-infrared detector arrays. Aspects of this software were shared with Prof. Howard, PI of another ALERT project.

V. REFERENCES

*** Indicates publications by senior personnel that are relevant to this project (Total: 6)**

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