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 to use the device for the stand-off detection of trace explosives in the solid phase. The proposed MIR-PIC is a mid-infrared (mid-IR) heterodyne receiver comprising a high-performance, mid-IR quantum cascade laser (QCL) with an integrated Schottky barrier diode. 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 novel, compact 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, 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 ultra-compact (~5 mm x 300 µm), low-cost, appropriate for commercial-scale production, and 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.

This project addresses a near-ubiquitous limitation of stand-off detection using mid-IR QCLs, including the relative intensity noise (RIN) of the lasers. The RIN of QCLs is most often the limiting factor in sensor performance and detection sensitivity. This project improves the sensitivity by orders of magnitude (approaching the quantum limit) by shifting the signal into the radio frequency (RF) domain via nonlinear mixing in the integrated diode; in the RF domain, the RIN of the QCL is substantially lower. Additionally, the integrated...
diode allows the MIR-PIC to function as both source and detector, enabling the ultra-compact footprint while reducing complexity and cost.

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 ultra-compact device. This research will have significant impact on the Department of Homeland Security (DHS) 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. Additionally, a myriad of other fields, including medicine, drug enforcement, and environmental monitoring, will also benefit.

B. Year Two (July 2014 through June 2015) Biennial Review Results and Related Actions to Address

This project is new to ALERT, and therefore was not part of the Biennial Review in Year 2.

C. State of the Art and Technical Approach

Optical spectroscopy has played an increasing role in materials characterization and sensing over the past two centuries. The growing role of light in characterization and detection applications has been enabled by our access to and control over different regions of the electromagnetic spectrum. By monitoring the interaction of light with matter, it is possible to gain qualitative and quantitative information about a sample under test [1]. Vibrational spectroscopy is concerned with interactions between light and vibrational and rotational states of molecules, which have unique spectral fingerprints that can be differentiated using these techniques. Mid-IR light interacts with the fundamental vibrational and rotational modes of many molecules, meaning that these light-matter interactions can be orders of magnitude larger than in neighboring spectral regions [2, 3]. Prior to the demonstration of mid-IR semiconductor lasers, most mid-IR spectroscopy was performed using expensive and bulky Fourier transform infrared spectrometers (FTIRs) or dye lasers, drastically limiting access to this portion of the spectrum. The QCL unlocked the mid-IR to the broader scientific and engineering communities, and enabled countless new applications—including explosives detection—across a myriad of fields.

Mid-IR QCLs have matured tremendously since their first demonstration in 1994 [4]. These electrically-injected devices emit mid-IR radiation via engineered optical transitions for electrons in semiconductor heterostructures. QCLs are now capable of room-temperature operation in pulsed and continuous wave (CW) mode, with optical output powers greater than 1 W [5]. Much of this progress has been enabled by careful quantum engineering of the semiconductor heterostructure comprising hundreds of coupled quantum wells. The lasers are available commercially from several domestic and international companies including Thorlabs Inc. [6], Daylight Solutions [7], and Alpes Lasers [8].

Despite their maturity and commercial availability, large gains in performance were recently realized by more carefully considering the physics of electron transport in the quantum well superlattice and revisiting the design of the QCL active region (i.e. the portion of the QCL responsible for generating gain). In these so-called "ultra-strong coupling lasers," the gain provided by the active regions is many times larger than more conventional QCL active region designs [9, 10]. These new designs with improved gain have resulted in lasers with record wall-plug efficiency (electrical-to-optical conversion efficiency) and optical powers [10]. In addition to improved wall-plug efficiency and high output powers, the improved gain can also be used to enable lasing in devices with novel waveguides, such as those employed by the MIR-PICs in this project, that might have larger optical losses than conventional waveguides.

QCLs have already been employed in a number of sensing setups. The most straight-forward incorporation of QCLs into infrared absorption spectroscopy (IAS) uses the QCL as a mid-IR source and a conventional de-
tector, typically HgCdTe (mercury cadmium telluride (MCT)), for monitoring the intensity of the mid-IR light after interacting with a sample under test [11]. More sophisticated sensing systems that improve the sensitivity and selectivity of the system have also been employed. These more sophisticated approaches include photoacoustic spectroscopy [12], cavity ringdown spectroscopy [13, 14], and Faraday rotation spectroscopy [15, 16], among others. These schemes have been used by a number of groups to detect explosives and precursors to explosives in the solid and gas phases [11, 17, 18, 19]. The detection sensitivity limit in all of these approaches is determined in part by fluctuations of the intensity of the QCL, that being the RIN.

The RIN noise is determined by a number of factors, including the current source and the underlying physics of the laser. Even for extremely stable current sources, the RIN of a QCL is larger compared to more traditional interband diode lasers due to the cascading of electrons in the active core. It has been demonstrated for QCLs that non-radiative losses out of the upper laser states dominate the RIN [20]. Since the RIN of the QCL is a fundamental impediment to the lasing physics, new strategies are needed to circumvent this limitation and to further improve the detection limit.

The three primary strategies to reduce laser noise are: 1) heterodyne detection, 2) active laser intensity stabilization, and 3) balanced detection [21]. Active laser intensity stabilization requires custom equipment that increases the cost, complexity, and footprint of the sensor. Balanced detectors with differential amplifiers are only available in the mid-IR via custom design, and are therefore very costly. Heterodyne detection shifts the detection to the RF domain (about 14 GHz for the devices in the project), where the QCL RIN is much lower [21]. Heterodyne detection using QCLs has been demonstrated using discrete free-space optical components [21, 22]. For heterodyne-enhanced Faraday rotation spectroscopy, systems have been demonstrated at only 5.6 times the ultimate theoretical sensitivity, demonstrating the significant improvements that can be realized using heterodyne detection.

In addition to improving the sensitivity by reducing RIN, heterodyne detection also enables acquisition of both phase and amplitude information for detected scattered or transmitted light without the need for calibration [21, 22 23]. Phase sensitive detection or imaging is often used to distinguish between similar materials. In the context of explosives detection, it may be possible to use phase sensitive detection to sort out decoys from true threats. Furthermore, combined phase and amplitude information can improve the sensitivity and selectivity of detection.

Unfortunately, with all the benefits of heterodyne detection, the size and complexity of the system is a significant drawback. Numerous free-space optical elements, such as lenses, mirrors, and acousto-optic modulators, are required to implement the technique. Our project advances the state of the art through the development and demonstration of PICs that combine a high-performance QCL active region with a diode engineered for heterodyne mixing of the internal modes of the QCL with back-scattered light that has interacted with a sample under test. Our MIR-PIC is a single semiconductor device that serves as both source and detector.

Figure 1 on the next page 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). The active core of the QCL generates mid-IR photons via electron transitions between the bound subbands 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, and re-enter the waveguide. These photons are then mixed by the Schottky diode with the internal field (photons) of the waveguide. 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) [24, 25]. For cavities ~ 3mm long, the IF is ~14 GHz, a frequency that is easily accessible using common microwave equipment available in our laboratories.
In the second year of this project (Year 5), we will use the developed and characterized MIR-PICs to demonstrate detection of trace explosives. We will focus on trace samples of C-4 (91% research department explosive (RDX)) already available in our laboratory. These samples are deposited on bare car-body aluminum, car-body aluminum with paint and clear coat, and low-reflectivity plastic. The trace amounts are deposited using gloved thumbprints and are provided by Prof. Steve Beaudoin at Purdue University (projects R1-A.1 and A.3). These explosives are already well-characterized in the mid-IR and will serve as an excellent benchmark for the performance of our MIR-PICs.

We will explore two configurations for stand-off detection: (1) with an external ZnSe lens, and (2) without an external lens. An external lens is useful for collimating the emitted beam and collecting more of the back-scattered light. Collimating the beam provides more power on target and collecting more light will improve the sensitivity of the MIR-PIC. We anticipate that including the external lens will improve the stand-off distance. While the external lens introduces an external optical component, this could be integrated into the final MIR-PIC package; this is not true for the optical components used in stand-off detection with discrete sources and emitters. We anticipate that for small stand-off distances appropriate for scanning fingerprints, handled documents, baggage, etc., an external lens is not needed.

We will systematically study stand-off detection using the MIR-PICs, including how the following parameters affect the sensor: (1) incident angle on the sample, (2) sample substrate, (3) stand-off distance, 4) operation mode of the LO (pulsed versus continuous wave and fixed-frequency versus tuning), (5) operating temperature, (6) LO power, (7) design and fabrication variations (cavity length, ridge width, diode design, diode location, facet coatings, etc.), (8) heterodyne integration time and amplifier chain, (9) LO tuning rate, and more. We will determine the MIR-PIC sensitivity by measuring the heterodyne signal versus the amount of solid trace explosive for a fixed integration time (1 s). For all measurements, we will compare the results with those obtained using a custom vacuum FTIR with a cooled HgCdTe detector that is available in our lab to verify that the measured heterodyne signal is from the trace explosive.

D. Major Contributions

In the first six months of this project, we have made excellent progress, including a close study of the preliminary MIR-PICs, the design of a new MIR-PIC active region and waveguide for the detection of RDX, and significant progress in the fabrication of the new MIR-PICs in the Notre Dame Nanofabrication Facility (NDNF). The following sections discuss these efforts in detail.

D.1. Study Preliminary Devices

We have fabricated and characterized MIR-PICs that exhibit a heterodyne signal from 14 GHz (3 mm cavity)
to ~21 GHz (2 mm cavity). A top view of a fabricated device is shown in Figure 2(a), and an exemplary diode response is shown in Figure 2(b). These preliminary measurements indicate that mixing in the Schottky diode is possible at mid-IR frequencies. However, while these preliminary data indicate the heterodyne detection is possible, the IF amplitude should be increased to improve the signal-to-noise ratio (SNR) and detection limit of the MIR-PIC. The IF amplitude can be improved via several approaches, including increasing the internal field of the MIR-PIC, redesigning the waveguide, and engineering the Schottky diode. Additionally, the tested devices only operate in pulsed mode. This is problematic since there is frequency chirp (i.e. a shift in the lasing frequency) over the duration of the applied pulse (200 nm to 2 µs in duration) due to heating of the active core. Our analysis of these initial devices indicates that a new, high-performance QCL active region with improved gain is necessary.

D.2. High-Performance QCL Active Core

The IF amplitude and SNR are related to the internal field of the MIR-PIC waveguide. From our preliminary measurements, we believe that the IF amplitude is limited by the internal field of the waveguide at the Schottky diode. To increase the internal field, we designed a QCL active core that incorporates elements from state-of-the-art, high-performance mid-IR QCLs. The active region is designed for detecting RDX at 6.25 µm and is depicted in Figure 3 on the next page. In addition to increasing the internal field, a high-performance design will also enable continuous wave operation, which is needed to avoid frequency chirp observed in pulsed mode.
For the design, we modified existing density matrix models in our laboratory to properly include coherent coupling and interface roughness scattering in the QCL. We have submitted a manuscript to the *Journal of Optics* related to these design efforts [26]. Compared to a conventional design at the same wavelength, our design is expected to exhibit gain more than five times larger. Figure 4 shows the results of calculations of the QCL gain versus the coherent coupling between the upper-laser level and the electron injection level. By increasing coherent coupling between these levels, we improve electron injection into the optical transition and increase gain. Increasing the coupling too much would lead to spectral splitting of the optical transition and a reduction in the peak gain. Importantly, these models could be used for designing other high-performance QCLs for ALERT projects or for projects with our external collaborators.

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**D.3. Increasing Nonlinear Mixing**

We also redesigned the waveguide of the MIR-PIC to increase the magnitude of the electrical field at the
contact of the laser, which will ultimately increase the non-linear mixing on the diode. We did this because our analysis shows that it should be possible to use the entire top contact of the laser as the Schottky diode, greatly simplifying the nanofabrication process. Since the waveguide we are employing is different than what is conventionally used for QCLs, we had to modify our custom design software. The redesigned waveguide and calculated field profile are shown in Figure 5.

Samples with the active region and waveguide designs shown above were grown via molecular beam epitaxy (MBE) through an unfunded collaboration with Princeton University. The samples were processed into standard QCL ridge waveguide lasers and are in the process of being characterized in Prof. Hoffman’s laboratory. The characterization includes spectral measurements to determine the lasing wavelength and light-current-voltage measurements, as means of ascertaining the electrical and optical properties of the lasers, such as peak power, threshold current density, temperature performance, etc. After the standard QCL characterization is completed we will fabricate MIR-PICs using the same semiconductor material and characterize the devices in the microwave domain. Dr. Wanke’s laboratory at SNL is already configured to measure these devices. Prof. Hoffman will send the graduate student working on this project to Sandia to be trained on RF measurements of MIR-PICs. The setup will be duplicated at Notre Dame using equipment available in our laboratory.

E. Milestones

This is a new project that began in January 2017. Since the start of the project, we have made significant progress towards the goals set in the Year 4 work plan, which focused on the design, fabrication, and characterization of MIR-PICs for this program.

- A new graduate student was recruited into the group to work full-time on this project. The student has a strong background in numerical modeling and simulation and is versed in both MATLAB and Python, the two software environments where our quantum and electromagnetic models are implemented.
- We have developed a numerical code to model the quantum and optical properties of the MIR-PICs. Using these models, we have designed MIR-PIC devices that aim to address problems with the devices used for our preliminary study, which were prevented from operating in continuous wave mode. Specifically, we have developed a quantum cascade gain region capable of providing double the gain of our previous de-

Figure 5: Waveguide design and the calculated mode intensity. At the Schottky diode/contact (shaded gold region), the mode intensity is about 30% of the peak mode intensity to increase nonlinear mixing on the diode.
sign. These new active region designs employ ultra-strong coupling to improve the injection of electrons into the upper-laser level.

- We have used our optical models to design new optical waveguides, focusing on optical loss, field strength at the metal contact, and the confinement factor. We have 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 have fabricated QCL devices and are in the process of performing standard electrical and optical characterization.

In Year 5 we will characterize the MIR-PICs in the microwave domain and demonstrate sensing using the MIR-PICs. These goals are summarized in the following objectives:

- Demonstrate room-temperature operation of MIR-PICs with >10x improvement in the IF amplitude SNR over preliminary devices.
- Demonstrate continuous-wave operation of the MIR-PIC and characterize improvement in IF amplitude SNR.
- Demonstrate stand-off detection of a trace explosive or mimic in the gas or solid phases using a MIR-PIC with a detection limit that is comparable to existing technologies that use discrete components. We are targeting a detection limit of better than 1 µg/cm² of C-4.
- Characterize stand-off detection on various substrates such as wood, cardboard, cloth, metals, painted/coated metals, etc.

**F. Future Plans**

In Year Five, we will achieve the following milestones:

- Characterize the MIR-PICs in the microwave domain.
- Demonstrate sensing using the MIR-PIC.
- Relate the sensor performance to MIR-PIC optical and electrical properties.

Programmatic risks include: (1) difficulties with material growth, and (2) low-yield device yield due to fabrication issues. The semiconductor heterostructures used for the devices in this program will be grown via MBE. MBE growth reactors are sophisticated machines capable of growing individual atomic layers of materials. As such, the machines often require scheduled and unscheduled maintenance that can last weeks to months, delaying the delivery of materials. To mitigate delays due to material delivery, we have identified two MBE labs at SNL and one at Princeton University that are capable of growing the heterostructures needed for this work. We will work with the individual who has grown preliminary devices for us but, if needed, we will shift our resources to engage our alternative grower. In addition to growth, the MIR-PICs in this proposal will need to be fabricated using a variety of nanofabrication tools. Challenges with nanofabrication include instrument downtime and device yield issues. Drs. Hoffman and Wanke have over 30 years of combined nanofabrication experience, and will use this experience to mitigate problems related to fabrication. For example, we will base the fabrication of the MIR-PIC on the fabrication of mid-IR QCLs, which have been fabricated at both Notre Dame and SNL.

Beyond Year 5, we are interested in improving the sensitivity of these integrated circuits, thus establishing a new gold-standard for explosives detection in the mid-IR. This new work would include: (1) optimizing the RF and optical design of the MIR-PIC; (2) implementing an external, highly-tunable optical source for broad
spectral coverage; (3) integrating nanophotonic elements on the facet of the MIR-PIC to enable stand-off detection for large distances (meter-scale) without any free-space optical components; and (4) packaging the MIR-PICs into real-world devices. The selected work plan and end date depend on the final Year 5 results as well as the needs of ALERT and DHS. These additional phases of research will improve the sensitivity and usability of the MIR-PIC. This work would also lower the detection limit of the device and reduce image acquisition times. Our goal for these additional phases of research are to: (1) improve MIR-PIC performance, (2) demonstrate clear advantages of MIR-PIC sensors, and (3) implement the technology in real-world applications and scenarios. These goals address DHS's need for highly-compact sensors capable of sensitive detection and imaging of explosives.

III. RELEVANCE AND TRANSITION

A. Relevance of Research to the DHS Enterprise

This project addresses the need for ultra-compact, sensitive sensors for the stand-off detection of trace explosives in the solid phase (ng/cm²). 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. Longer stand-off distances should be possible and can be investigated if additional time is allotted for this project.
- 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/cm²; 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 ultra-compact footprint could enable handheld deployment.

B. Potential for Transition

Our vision for this technology within the DHS enterprise is to develop a linear array of MIR-PICs for rapidly scanning 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. Outside of the DHS enterprise, we envision that this technology will be used for imaging the margins of tumors during breast cancer surgery. For such an application, rapid imaging is needed to determine if the entire tumor has been removed from the breast tissue. We are collaborating on this application with Dr. Jennifer Tseng M.D., Fellow of Surgical Oncology at the University of Chicago, and plan to apply for funding from the National Institute of Health once we have preliminary data.

C. Data and/or IP Acquisition Strategy

Possible IP and disclosures are in the area of device and use patents. We are working with Tim Joyce in the Technology Transfer Office at Notre Dame to disclose potential IP and to submit patent disclosures and applications as necessary.
D. Transition Pathway

This project involves close collaboration with SNL, including student internships, as a direct path to transitioning this technology (and graduated students) to DHS customers. Year 5 includes a student internship at SNL. A student has already been identified to work with Dr. Wanke on device characterization in the microwave domain.

Ultimately, we aim to develop a MIR-PIC that can be transitioned for wide-scale deployment. To this end, we are pursuing a collaboration with Dr. Loan Li, Ph.D. of Northrup Grumman, who has expressed interest in the MIR-PICs being developed through this research. Our contact is interested in the possibility of developing mid-IR ultra-compact sensors based on our MIR-PICs. We also have a collaborative relationship with Dr. Yamac Dikmelik, Ph.D., at Thorlabs Inc., a leading company in mid-IR optoelectronics. Dr. Yamac is interested in the impact our photonic integration could have on their existing line of QCL products. We are submitting a journal article for publication, and our contact at Thorlabs is a coauthor on that manuscript [26]. The ultimate transition products also include students trained in technical areas relevant to the Homeland Security Enterprise (HSE), in addition to the MIR-PICs and derivative technologies developed in this program.

E. Customer Connections

Dr. Yamach Dikmelik, Ph.D., Thorlabs Inc.; Frequency of contact: Every other month; Level of involvement: We are submitting a manuscript with Dr. Yamac as a coauthor and collaborate with him on QCL design.

Dr. Loan Li, Ph.D., Northrup Grumman; Frequency of contact: infrequent; Level of involvement: Has expressed interest in our work and a possible project with funding. Funding is not available at the moment due to budgetary priority issues within Northrup Grumman.

IV. PROJECT ACCOMPLISHMENTS AND DOCUMENTATION

A. Education and Workforce Development Activities

1. Student Internship, Job, and/or Research Opportunities
   a. Hosted two undergraduate students through the University of Notre Dame Undergraduate Research Fellowship (NURF) Program (June 2017).

2. Interactions and Outreach to K-12, Community College, and/or Minority Serving Institution Students or Faculty
   a. All six graduate students in Prof. Hoffman’s group were involved in an outreach event with Clay Middle School that is related to the research in this project (see Fig. 6 on the next page). The outreach project was organized by a third-year graduate student, Owen Dominguez, and introduced students to light and spectroscopy through student participation in different modules during a day-long event. Approximately 100 7-9th grade students participated in the event. The event included hands-on demonstrations and activities and short discussion sessions. The students learned about fundamental concepts in optics, such as polarization, reflection and transmission, total internal reflection, lasing, and imaging.

   For our visit to Clay Middle School, we developed a new module where the students learned about mid-IR light and its applications. As part of the new module, the students had the opportunity to have their picture taken using a mid-IR camera. Figure 7 on the next page is a scaled reproduction of a 4x6” photograph that was given to each student as a souvenir.
B. Peer Reviewed Journal Articles

Pending-

C. Other Presentations

1. Seminars


V. REFERENCES


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