

international workshop on opportunities and challenges in mid-infrared laser-based gas sensing

15 - 17 May 2017 Wrocław, Poland

**Book of abstracts** 



4<sup>th</sup> International Workshop on Opportunities and Challenges in Mid-Infrared Laser-Based Gas Sensing

15 - 17 May 2017 Wrocław, Poland

### Support

This workshop is supported by *iCspec* project that has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 636930.

### Venue

The Congress Center (building D-20) on the campus of the Wrocław University of Science and Technology, Wrocław, Poland

### Mirsens workshops

Mirsens 4 continues the tradition of previous workshops of the series, which took place in 2010 and 2012 in Wrocław, and in 2015 in Würzburg. Its main goal is to promote interaction between academic and industrial institutions active in MIR research worldwide, and to address scientific and technological challenges and application prospects associated with this field.

Mirsens 4 is organized within iCspec Project (http://www.icspec.eu/) of Horizon 2020 Programme of the European Commission by Wrocław University of Science and Technology (Department of Experimental Physics, Faculty of Fundamental Problems of Technology and Centre for Advanced Materials and Nanotechnology). The workshop is organized as a combination of plenary lectures given by world-renowned experts and short contributed talks, plus a poster session.

### The main topics of *mirsens 4* workshop:

- Semiconductor materials and structures for the MIR
- Recent progress in MIR laser sources
- Laser-based gas sensing and spectroscopic techniques
- New application prospects

### **Committees**

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		М	onday - 15.05.2017
8:00	Recep	tion desk open	
9:00	9:10	Opening Address	s
9:10	9:40	R. Strzoda	In-line Cascade Laser Spectrometer for Process Control - iCspec Project
		SESS	SION: Sensing systems I
9:40	10:20	P. Kaspersen	Laser-based Spectroscopy – A Success Story?
10:20	11:00	G. Wysocki	Interband Cascade Laser-Based Dual-comb Multi- Heterodyne Spectroscopy of Small and Large Molecules
11:00	11:20	Coffee break	
		SESS	ION: Sensing systems II
11:20	11:40	F. Tittel	Recent Advances and Applications of Mid-infrared Cavity and Quartz Enhanced Photoacoustic Spectroscopy
11:40	12:00	P. Kluczyński	Multi-component Mid-IR Tunable Laser Analyzers for Process Control
12:00	12:20	K. Krzempek	Photothermal Spectroscopy of NO at 5.2 µm Using Quantum Cascade Laser and Near-infrared Heterodyne-based Detection
12:20	12:40	J. Waclawek	2f-Wavelength Modulation Fabry-Perot Photothermal Interferometry
12:40	13:00	A. Hudzikowski	Compact, Low Power Mid-infrared Methane Isotope <sup>13</sup> CH <sub>4</sub> and <sup>12</sup> CH <sub>4</sub> Sensor Using Room-temperature CW Interband Cascade Laser (ICL)
13:00	14:00	Lunch break	
		SESSION:	Detectors & sources in MIR I
14:00	14:40	MC. Amann	Single-mode Tunable VCSELs for the 2-4 µm Wavelength Range
14:40	15:20	J. Piotrowski	Recent Progress in Development of Mid-IR Detection Modules for Gas Analyzers
15:20	15:40	Coffee break	
		SESSION:	Detectors & sources in MIR II
15:40	16:00	F. Kapsalidis	Stable, High-Power Quantum Cascade Laser Frequency Combs Operating in Room Temperature
16:00	16:20	A. Pfenning	GaSb-based Resonant Tunneling Structures with Ternary Prewell Injectors for Room Temperature Mid- Infrared Applications
16:20		K. Pierściński	Analysis of Heat Dissipation Schemes in QCLs
16:40	17:00	V. Gramich	Type-II Superlattice Photodetector Developments in the Mid-Infrared Region
17:00	17:20	R. Wang	Widely Tunable 2.3 µm InP-based Type-II DFB Laser Array Heterogeneously Integrated on Silicon for Sensing
17:20	17:40	M. Motyka	Carrier Dynamics in GaSb-based Quantum Wells Emitting in the 2 µm Range
17:40	End of	the day	
19:30	Confe	rence Dinner in D	owntown

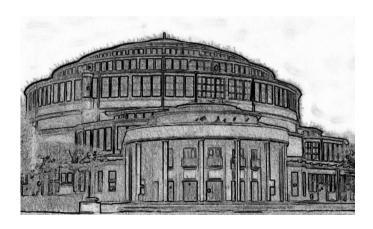
Tuesday - 16.05.2017			
		SESS	ION: Laser sources I
10:00	10:40	M. Bugajski	Strain-compensated AlInAs/InGaAs/InP Quantum Cascade Lasers
10:40	11:20	R. Yang	Recent Progress in Interband Cascade Devices
11:20	11:40	Coffee break	
		SESS	ION: Laser sources II
11:40	12:00	K. Abramski	Fully-fiberized Multi-wavelength Difference Frequency Generation Mid-infrared Source for Laser Spectroscopy Applications
12:00	12:20	M. Shahmohammadi	Dual-wavelength DFB Quantum Cascade Lasers for NO and $\mathrm{NO}_2$ Sensing
12:20	12:40	P. Gutowski	MBE Growth and Fabrication of $In_xGa_{1-x}As/Al_{0.45}Ga_{0.55}As/GaAs$ Strained Quantum Cascade Lasers
12:40	13:00	P. Moselund	Mid-infrared Supercontinuum – A Maturing Technology
13:00	13:20	T. Czyszanowski	Quantum-cascade Vertical-cavity Surface-emitting Laser
13:20	13:40	M. Gładysiewicz	Electronic Band Structure and Material Gain in GaSb- based Quantum Wells Containing Bismuth: Toward Enhancement of Quantum Confinement in the Valence Band
13:40	14:30	Lunch break	
14:30	15:30	Poster session	
		SESSIC	N: Sensing systems III
15:30	16:10	B. McManus	Recent Progress in Trace Gas Instrument Development at Aerodyne Research, Inc.
16:10	16:50	R. Kan	MIR TDLAS Technology for Industrial Emission and Environmental Monitoring
16:50	17:10	R. Heinrich	High Performance Spectroscopy of Hydrocarbon Gas Mixtures in the 6 – 11 μm Range
17:10	17:30	O. Aseev	Breath Alcohol – High Precision Measurement of VOCs Using a DFB-QCL
17:30	17:50	A. Ghetti	TDLAS Determination of Carbon Dioxide Isotope Ratio for Diagnosis of Helicobacter Pylori
17:50	End of	the day	

	Wednesday - 17.05.2017					
	SESSION: Sensing systems IV					
9:00	10:20	P. Geiser	In-situ $H_2S$ and $SO_2$ Tail Gas Analysis with Near- and Mid-infrared TDLS			
9:20	9:40	G. Dudzik	IQ Demodulation-based Gas Sensing Detection System for the Photothermal and Chirped Laser Dispersion Spectroscopy			
9:40	10:00	M. Graf	Compact and Lightweight Multipass Cell Designs with Optimized Beam Propagation			
10:00	10:20	L. Cocola	Temperature Measurements From 2 µm Carbon Dioxide Absorption Spectrum			
10:20	10:40	YP. Tseng	Upconversion Detection for Gas Sensing Applications			
10:40	11:00	T. Kääriäinen	Stable Isotope Analysis of <sup>13</sup> CH <sub>4</sub> and CH <sub>3</sub> D in Mixed Biogenic and Fossil Methane Samples			
11:00	11:20	Coffee break				
SESSION: Laser sources III						
11:20	11:40	Y. Bidaux	Waveguide Engineering for Low Dispersion Mid-infrared Quantum Cascade Lasers Frequency Combs			
11:40	12:00	G. Hałdaś	Tuning Quantum Cascade Laser Wavelength by the Injector Doping			
12:00	12:20	I. Šimonytė	GaSb SLDs and Gain-chips for Sensing Applications in the 2-2.5 Micron Wavelength Range			
12:20	12:40	E. Pruszyńska- Karbownik	Quantum Cascade Lasers with Nonuniformly Tapered Waveguides			
12:40	13:00	M. Dyksik	Triple Quantum Wells for Active Regions Of Mode- locked ICLs in the Mid-infrared			
13:00	13:20	S. Höfling	Interband Cascade Lasers on GaSb Substrates Emitting Beyond 5.6 $\mu m$			
13:20	14:00	Lunch break				
	SESSION: Laser sources IV					
14:00	14:40	J. Meyer	Advances in Interband Cascade Lasers and LEDs			
14:40	15:20	J. Koeth	Specialized Laser Sources for Sensing in the MIR			
15:20	15:30	Closing Remarks	S			
15:30	End of	the workshop				

# 15 May (Monday)

### Sessions

Sensing systems I
Sensing systems II
Detectors and sources in MIR I
Detectors and sources in MIR II



**Centennial Hall** 

### Opening presentation

# In-line Laser Cascade Spectrometer for Process Control – *iCspec* Project

### Rainer Strzoda

Corporate Technology, Siemens AG, Otto-Hahn-Ring 6, 81739 Munich, Germany

The mirsens 4 workshop is linked this time to the Horizon 2020 project iCspec<sup>1</sup> (*in-line Cascade laser spectrometer for process control*). Thus the first talk of the workshop shall introduce the project and draw the attention to the various presentations to be given by members of the consortium during the workshop. Further information about the project can be found on the project website: http://www.icspec.eu.

The basic idea behind the project is to build a laser spectrometer with a wide tuning range to allow the spectral measurement of line spectra as well as unresolved broad absorption bands. Laser arrays fulfill these requirements in principle. ICL-arrays in the NIR and QCL-arrays in the MIR range allow accessing the whole spectral range from 3 to 12  $\mu$ m. Within the project selected spectral ranges covering the target gas species will be realized to demonstrate multicomponent gas analysis (Fig.).

In a pilot application the performance of multi-gas analysis shall be demonstrated for the first five hydrocarbons Methane, Ethane, Propane, Butane and Pentane. In the distillation process of crude oil in a refinery the continuous instantaneous measurement of the composition of the gas fraction is a long demanded requirement. Gas chromatographs as the state of the art instrumentation today suffer from delay times of several minutes preventing real time process control leaving optimization potential unused.

The project comprises three areas, the development of the light sources, the analyzer development and the field test. The light sources integrated into a butterfly package consist of the laser-array chip, an ASIC for driving the lasers individually and a beam combiner. The analyzer development includes the determination of the not yet available high resolution reference spectra of the heavier hydrocarbons, the data analysis and the development of two demonstrator gas analyzers. The field test of the demonstrator instruments will be carried out on site in a refinery.

In the talk the general concept as well as examples of the progress in the project so far will be presented.

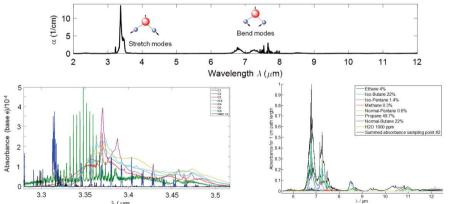


Fig. Hydrocarbon spectra of the target gases in the  $2-12 \mu m$  accessible with ICL- and QCL-arrays.

<sup>&</sup>lt;sup>1</sup> The project has received funding from the European Unions Horizon 2020 research and innovation program under grant agreement No 636930.

### INVITED

### Laser-Based Spectroscopy - A Success Story?

### Peter Kaspersen<sup>1</sup>, Peter Geiser<sup>2</sup>

Norsk Elektro Optikk AS, Prost Stabels vei 22, 2019 Skedsmokorset, Norway
 NEO Monitors AS, Prost Stabels vei 22, 2019 Skedsmokorset, Norway

If you are being asked "Is laser-based spectroscopy a success story?", many will answer "No doubt, it is!". But is it really? Let's have a closer look.

Two years after the first demonstration of laser emission in 1960 [1], a key element to laserbased absorption spectroscopy was invented; semiconductor lasers emitting in the nearinfrared region [2]. Huge investments by the telecom industry in the 1970s and 80s made these lasers a mass-product. Fortunately, many gases have overtone absorption bands in the same wavelength region, so this enabled tunable diode laser spectroscopy (TDLS) to become more widespread. First companies were founded (e.g., Norsk Elektro Optikk and AltOptronic) pioneering application fields like in-situ industrial gas sensing. At that time, the first success stories were beginning. In the course of time, new laser technologies emerged covering different wavelength regions. Especially cascade laser technologies were a breakthrough starting in the mid-1990s [3,4], making the fundamental absorption bands in the mid-infrared available allowing the measurement of important gases like sulfur dioxide in sulfur recovery plants or tetrafluoromethane in the Aluminum industry [5]. More and more companies were sprouting like mushrooms and started their own success stories (e.g., Cascade Technologies, AirOptics ...). Aside from measurements in industrial environments, other application fields were conquered as well. Medical applications, for example, are getting more and more into focus of interest monitoring breath for diagnostic purposes [6] or even inspecting lungs of preterm babies [7]. Waiting in the starting blocks is now the extension of the technology to even longer wavelengths, the THz range, by trying to develop lasers using new materials like Graphene.

In addition to the commercial exploitation of applications and development of new lasers, the scientific community investigated new detection schemes achieving incredible sensitivities and compactness (e.g., QEPAS [8]).

But not only the gas phase is of interest, also the liquid phase comprises many interesting applications exploited by companies like *QuantaRed*.

In recent years, big players in the analytical market became aware and interested in laser-based spectroscopic sensors and first acquisitions started indicating a wide acceptance and maturity of the technology. Small companies were integrated into bigger ones, which is usually a good sign of success.

So, coming back to the initial question, the answer should be "Yes! Though it is not one but many success stories. However, the search for the big one, the real mass-market product is still in progress."

- [1] T. H. Maiman, *Nature* **187**, 4736, pp. 493-494 (1960)
- [2] R. N. Hall et al, *Phys Rev Lett* **9**, 366 (1962)
- [3] J. Faist et al, *Science* **264**, pp. 553-556 (1994)
- [4] R. Q. Yang, Superlatt Microstruct 17, pp. 77-83 (1995)
- [5] P. Geiser et al, *Photonics* **3**, 16 (2016)
- [6] C. Wang et al, Sensors 9, pp. 8230-8262 (2009)
- [7] NEO-Lung project: https://www.forskningsradet.no/prosjektbanken/#!/project/251799/en
- [8] A. A. Kosterev et al., Opt Lett 27, 1902 (2002).

# Sensing Systems

# Interband Cascade Laser-Based Dual-Comb Multi-Heterodyne Spectroscopy of Small and Large Molecules

J. Westberg<sup>1</sup>, L.A. Sterczewski<sup>1,3</sup>, L. Patrick<sup>1</sup>, C.S. Kim<sup>2</sup>, M. Kim<sup>4</sup>, C.L. Canedy<sup>2</sup>, W.W. Bewley<sup>2</sup>, C.D. Merritt<sup>2</sup>, I. Vurgaftman<sup>2</sup>, J.R. Meyer<sup>2</sup>, and G. Wysocki<sup>1\*</sup>

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<sup>2</sup>Naval Research Laboratory, Code 5613, Washington, DC 20375, USA (\*e-mail: gwysocki@princeton.edu)
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Laser spectroscopy has proven to be a one of the most versatile as well as sensitive and selective chemical detection technologies that can address a broad variety of applications such environmental monitoring, industrial emission control, or security and safety. Mid-infrared molecular fingerprint region is one of the most important spectral domains that enables highly sensitive and selective chemical detection. Therefore there is a need for mid-IR laser sources and spectroscopic techniques that could enable simultaneous detection of a multitude of important molecular species with high spectral resolution. This generally requires broadband optical bandwidth that in addition to multispecies detection can also enable detection of more complex molecules with broader spectral features. There are several technologies that attempt to address this application domain that includes: mid-IR external cavity lasers [1], which provide large spectral coverage obtained by opto-mechanical tuning, or recent advancements in stabilized frequency combs with nonlinear frequency-conversion [2] to micro-resonator frequency combs in the mid-infrared [3] that provide broadband frequency coverage. Unfortunately, these systems are typically quite complex and/or vibration sensitive, which limits their potential to be used in field applications.

Mid-IR semiconductor laser technologies such as quantum- and interband cascade lasers (ICLs) have recently become common off-the-shelf devices available from several manufacturers. Especially ICLs are characterized by high wall-plug efficiency and low threshold electrical power that make them particularly attractive for power-constrained sensing applications. To achieve simultaneous broadband coverage combined with high spectral resolution it has been recently demonstrated that multiheterodyne spectroscopy [4] using mid-infrared Fabry-Pérot lasers can be reliably performed [5-9]. The multiheterodyne process provides a truly parallel down-conversion of optical spectroscopic information acquired by all laser modes to RF domain, where the signals can be conveniently acquired and analyzed. In this work we present capabilities of multi-heterodyne spectroscopy implemented with Fabry-Perot ICLs. The ultimate spectral resolution is limited by the cumulative linewidth of free-running ICLs, which resulted in multi-heterodyne beat note linewidth of ~800 kHz. We also developed computational phase and timing correction algorithms which assume frequency comb-like behavior of the ICLs, and provide further reduction in the linewidth of the beat notes down to the Fourier limit of ~2kHz. To demonstrate the applicability of these light sources to broad spectral gas sensing, proof-of-concept measurements of methane and ethylene around 3.21 μm were performed with time resolution down to 20 μs. Most recent research progress and performance tests of ICL-based multi-heterodyne system will be discussed in details.

**Acknowledgments** The work at Princeton was supported by the DARPA SCOUT program (W31P4Q161001). The work at NRL was supported by ONR.

- R. F. Curl, F. Capasso, C. Gmachl, A. A. Kosterev, B. McManus, R. Lewicki, M. Pusharsky, G. Wysocki, and F. K. Tittel, "Quantum cascade lasers in chemical physics," Chem Phys Lett 487, 1-18 (2010).
- [2] I. Coddington, N. Newbury, and W. Swann, "Dual-comb spectroscopy," Optica 3, 414-426 (2016).
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- [4] S. Schiller, "Spectrometry with frequency combs," Optics Letters 27, 766-768 (2002).
- [5] Y. Wang, M. G. Soskind, W. Wang, and G. Wysocki, "High-resolution multi-heterodyne spectroscopy based on Fabry-Perot quantum cascade lasers," Appl Phys Lett 104, 0311141-0311145 (2014).
- [6] G. Villares, J. Faist et. al, "Dual-comb spectroscopy based on quantum-cascade-laser frequency combs," Nature Comm. 5, 5192 (2014).
- [7] Y. Yang, Q. Hu et. al, "Terahertz multiheterodyne spectroscopy using laser frequency combs," Optica 3, 499-502 (2016).
- [8] A. Hangauer, J. Westberg, E. Zhang, and G. Wysocki, "Wavelength modulated multiheterodyne spectroscopy using FP-QCLs," Opt. Expr. 24, 25298-25307 (2016).
- [9] L.A. Sterczewski, J. Westberg, and G. Wysocki, "Molecular dispersion spectroscopy based on FP-QCLs," Opt. Lett. 42, 243-246 (2017).

### MoO<sub>1</sub>

# Recent Advances and Applications of Mid-infrared Cavity and Quartz Enhanced Photoacoustic Spectroscopy

F.K. Tittel  $^1$ , V. Spagnolo  $^{1,2}$ , P. Patimisco  $^{1,2}$ , M. Giglio  $^{1,2}$ , A. Sampaolo  $^{1,2}$ , W. Ye $^1$ , Q. He $^1$ , H. Zheng  $^{1,3}$ , M. Lou  $^1$ 

<sup>1</sup> Department of Electrical and Computer Engineering, Rice University, 6100 Main Street, Houston, TX 77005, USA

The recent development of compact interband cascade lasers (ICLs) and quantum cascade lasers (QCLs) capable of targeting strong fundamental rotational-vibrational transitions in the mid-infrared has led to the design and fabrication of mid-infrared compact, field deployable sensors based on cavity absorption and enhanced photoacoustic spectroscopy. These sensors have found use in the petrochemical industry, environmental monitoring, monitoring, atmospheric chemistry, life sciences, medical diagnostics, defense and security applications. Specifically, the spectroscopic detection and monitoring of four molecular species, methane (CH<sub>4</sub>), ethane (C<sub>2</sub>H<sub>6</sub>), formaldehyde (H<sub>2</sub>CO) and hydrogen sulphide (H2S) will be described.

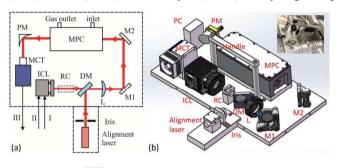
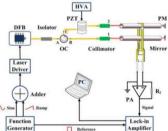


Fig. 1. Schematic of the mid infrared cavity sensor, CAD image of sensor reduced dimensions of length (35.5 cm), width (18 cm), and height cm). Inserted image: photograph of sensor core. ICL: interband cascade laser; DM: dichroic mirror; L: lens; M: plane mirror; MCT: mercury-cadmium-telluride detector; MPC: multi-pass gas cell; PM: parabolic mirror; RC: reference cell.



**Fig. 2.** Schematic of a double antinode excited quartz-enhanced photoacoustic spectroscopy apparatus. DFB: distributed feedback diode laser; OC: optical circulator; PZT: piezoelectric transducer; HVA: high voltage amplifier; PM: power meter; PA: pre-amplifier; and PC: personal computer.

[1] L. Dong, F. K. Tittel, C. Li, N. P. Sanchez, H. Wu, C. Zheng, Y. Yu, A. Sampaolo, and R. J. Griffin, *Optics Express*, **24**, A51 (2016).

[2] H. Zheng, L. Dong, P. Patimisco, H. Wu., A. Sampaolo, X. Yin, S. Li, W. Ma, L. Zhang, W Yin, L. Xiao, V. Spagnolo, S. Jia, and F. K. Tittel, *App. Phys. Letters*, **110**, 02110 (2017).

F.K. Tittel acknowledges the support from the Robert Welch Foundation (Grant C-0586), NSF ERC MIRTHE award, DOE ARPA-E, (DE-0000545, DE-0000547)

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# Multi-Component Mid-IR Tunable Laser Analyzers for Process Control

K. Siembab, J. Derezynski, M. Straszewski, J. Peziak, D. Luczak, S. Tomczyk, M. Suski, A. Wojcik and P. Kluczynski\*

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Airoptic develops and manufactures tunable laser analyzers for process control as well as safety and security applications. Airoptic's GasEye series analyzers are available in open path, extractive as well as cross duct *in-situ* (Figure 1) configurations. The main features are high selectivity and sensitivity, response times down to 50 ms, negligible zero drift and no field calibration thanks to a built-in reference gas cell and autocalibration features. Recently Tunable Laser Spectroscopy (TLS) has extended its capabilities into the mid-IR region offering many new opportunities for industrial as well as safety and security applications [1-3]. Until now, the lack of suitable laser sources in the main hydrocarbon absorption bands has limited the applicability of TLS in chemical industry. Tunable mid-IR laser sources in the wavelength range 3.3 – 7.0 μm, utilizing novel Interband Cascade Laser (ICL) structures, are now available This enabled development of novel photonic gas sensors for applications in process analytics and allowed to extend current portfolio of Near-IR based TDL analyzers to Mid-IR based devices.



**Fig. 1:** GasEye *in-situ* cross duct sensor for low ppb formaldehyde monitoring

In 2014, the company released the first Mid-IR laser based analyzer for formaldehyde monitoring, GasEye HCHO. The system is capable of detection of formaldehyde vapor in low ppb range directly in the process with response time below once second, see Figure 1. The system is mainly used in wood based panel

production as well as continuous real time monitoring of formaldehyde in work places. Recently, Airoptic has extended its offer towards multi-component analyzers, both in Near-IR and Mid-IR. Analysis of eight different components is currently possible with one analyzer. Typical examples are SO<sub>2</sub>/SO<sub>3</sub>, CO/H<sub>2</sub>O/CH<sub>4</sub> as well as H<sub>2</sub>O/H<sub>2</sub>S/CO<sub>2</sub> in natural gas for insitu and extractive configurations, respectively. One of the newest developments is a fast, sensitive and selective in-line multicomponent tunable laser analyzer for continuous monitoring of C1-C5 hydrocarbon composition in a process stream. The analyzer requires no consumables (e.g. purging, carrier gas) and no calibration in field, bringing the overall cost of ownership down. The C1-C5 analyzer has been developed within the European Commission framework program, iCspec [4], and is scheduled for field deployment this spring.

- P. Kluczynski, S. Lundqvist, S. Belahsene, Y. Rouillard, L. Nähle, M. Fisher, J. Koeth, "Detection of propane using tunable diode laser spectroscopy at 3.37 μm," Applied Physics B 108 (1), 183-188 (2012).
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- [4] http://www.icspec.eu/

### MoO<sub>3</sub>

# Photothermal Spectroscopy of NO At 5.2 µm Using Quantum Cascade Laser and Near-Infrared Heterodyne-Based Detection

### K. Krzempek<sup>1\*</sup>, M. Nikodem<sup>2</sup>, G. Dudzik<sup>1</sup>, G. Wysocki<sup>3</sup>, K. Abramski<sup>1</sup>

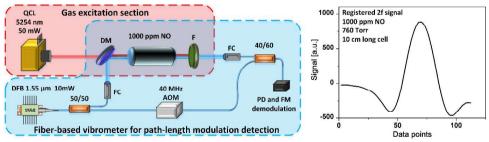
<sup>1</sup> Laser&Fiber Electronics Group, Wrocław University of Science and Technology, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Poland; email: karol.krzempek@pwr.edu.pl <sup>2</sup> Wrocław Research Centre EIT+, 54-066 Wrocław, Poland; <sup>3</sup> Princeton University, Princeton, NJ, USA

In this paper we present preliminary results on laser spectroscopy in the mid-IR wavelength range using photothermal effect with interferometric, heterodyne-based detection. The photo-thermal signal is induced when a gas sample is excited by a radiation with frequency matching the energy of the molecular transition. The absorption of radiation produces subtle, local increase of gas temperature and consequently a change of its refractive index. By analyzing these local variations the target analyte concentration can be determined.

In the proposed approach photo-thermal signal is detected using an optical interferometer. This heterodyne detection offers flexibility not available in typical photoacoustic-based systems. For example, the excitation source can be chosen independently from the probing source, allowing for exploiting their individual advantages. Moreover, modulation frequency can be freely chosen.

The presented setup (shown in Fig. 1) was designed to target nitric oxide (NO) particles enclosed in a gas cell. It can be divided into two separate parts - gas excitation source and path-length modulation detection. The excitation source was a 5.25 µm distributed feed-back (DFB) quantum cascade laser (QCL) with an output power up to 50 mW. The laser wavelength was swept across an NO absorption line at 5254 nm (1903.16 cm<sup>-1</sup>) by modulating the injection current with a ramp signal. Additional 500 Hz sine modulation was used to enable phase-sensitive detection of the path-length changes, corresponding to the induced photo-thermal refractive index variations. The second part of the sensor was constructed as a telecom-fiber-based laser Doppler vibrometer [1]. The emission from a 10 mW, 1550 nm DFB fiber-pigtailed laser diode was split via a 3dB coupler to form a reference and a probing arm of the interferometer. The probing beam was co-linearly combined with the emission of the QCL via a dichroic mirror and propagated through a 10 cm long gas cell filled with 1000 ppmv of NO at 760 Torr. The reference signal was frequency-shifted by 40 MHz with an acousto-optic modulator (AOM). After passing through the absorption cell the excitation beam was blocked with a filter while the probing beam was combined with the reference beam via a 40/60% coupler and detected by an InGaAs detector. The signal was demodulated using a custom-designed IQ demodulator and analyzed at 1 kHz (the second harmonic of the 500 Hz modulation). The photo-thermal refractive index changes induced in the NO gas sample, and consequently changes in the path-length seen by the laser vibrometer were recovered, creating a clear 2f signal of the targeted absorption line (shown in Fig. 1).

During the conference we will present our most recent results, including open-path photo-thermal detection. Potential new configurations and applications will be discussed.



**Fig. 1** System setup (left) and registered 2f photo-thermal signal of a 10 cm long gas cell filled with 1000 ppm of NO at 760 Torr (right). DFB – distributed feedback laser diode, FC – fiber collimator, DM – dichroic mirror, AOM – acousto-optic modulator, F – filter, 50/50 and 40/60 – fiber coupler, PD – photodiode.

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### Acknowledgments

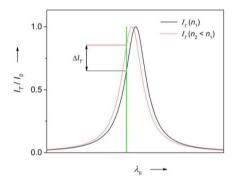
We acknowledge support from the National Science Centre within the project DEC-2014/14/M/ST7/00866.

# 2f - Wavelength Modulation Fabry-Perot Photothermal Interferometry

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Trace gas detection was performed by the principle of photothermal interferometry [1] using a Fabry-Perot interferometer (FPI) combined with wavelength modulation and second harmonic detection (2f) [2]. This arrangement enables the construction of highly compact and selective sensors having ultra-low absorption volumes. In this work, a quantum cascade laser was used as powerful mid-infrared excitation source to induce refractive index changes in the sample by molecular absorption, whereas a near-infrared laser diode served as probe source to monitor photo-induced variations (see Figure 1).



**Fig. 1.** Operation principle of the FPI based photothermal sensor: The frequency of a probe laser is tuned near to the inflection point of the periodic transmission function of the interferometer incorporating sample gas at thermal equilibrium (black trace). After a photo-induced heating of the sample by an excitation laser the refractive index of the gas decreases, which is accompanied by a shift of the transmission function with respect to the vacuum wavelength (red dotted trace). This shift is monitored by a change of the transmitted probe laser intensity.

The functional principle of the sensor was investigated by detection of sulfur dioxide (SO<sub>2</sub>) at an absolute pressure of 200 mbar. The FPI used as transducer for monitoring induced refractive index changes consisted of two dielectric coated mirrors with a reflectivity of 0.85, which were fixed by a distance of 1 mm to each other, yielding a gas cell with a total volume of < 0.7 cm<sup>3</sup>. For the targeted SO<sub>2</sub> absorption band centered at 1379.78 cm<sup>-1</sup> a 1  $\sigma$  minimum detection limit of about 1 parts per million by volume was achieved by employing a modulation frequency of 500 Hz and a lock-in time constant of 1 second. The work demonstrates the high potential for further sensor miniaturization down to a sample volume of only a few mm<sup>3</sup>. Limitations and possible improvements of the sensor regarding sensitivity are discussed.

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### MoO<sub>5</sub>

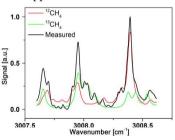
# Compact, Low Power Mid-infrared Methane Isotope <sup>13</sup>CH<sub>4</sub> And <sup>12</sup>CH<sub>4</sub> Sensor Using Room-temperature CW Interband Cascade Laser (ICL)

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Measurements of methane isotopic ratio is very important for environment. The level of gas in the atmosphere has significant impact on its radiative forcing, increase is estimated to 0.48 W/m<sup>2</sup> since pre-industrial times [1]. The origin of the gas can be obtained from isotopic ratio of carbon in CH<sub>4</sub>. Due to recent development of compact, low power interband cascade lasers (ICLs) in mid-infrared region, covering strong fundamental rotational-vibrational transitions absorption lines, at room temperature conditions, is possible to build compact, low energy consumption, field deployable gas sensors [3].

We report an experimental investigation of the performance of a compact gas sensor employing a 0.8 mW interband cascade laser (ICL) combined with custom made, innovative 24 m optical-path spherical gas cell with 80 ccm total volume. The sensor is developed to reach methane absorption lines at 3007.95 cm<sup>-1</sup> and 3008.39 cm<sup>-1</sup>, corresponding to <sup>13</sup>CH<sub>4</sub> and <sup>12</sup>CH<sub>4</sub> respectively. WMS was performed with custom made, ultra-low noise current source, custom acquisition card and a sensitive Vigo detector. Control signals and conditioned data are processed by low power 32-bits microcontroller with FPU (Floating Point Unit). The most suitable absorption lines of methane isotopes are located between 3003 – 3009 cm<sup>-1</sup>. The strongest line of <sup>13</sup>CH<sub>4</sub> is located at 3007.95 cm<sup>-1</sup> and the <sup>12</sup>CH<sub>4</sub> line with similar absorption is adjacent. A triangle signal was added to perform modulation of the ICL current, it sweeps between 3007.6 – 3008.7 cm<sup>-1</sup> and collect both isotopes absorption lines. The proposed sensor can achieve a precision below 0.3‰ of isotopic ratio for a 200 ppm methane mixture, which is better than previously reported precision [4]. **Figure 1** presents the directly acquired signal of 200 ppm methane mixture.



**Fig. 1.** Acquired absorbance spectra (black line) of 200 ppm methane mixture with simulated Hitran database plot for isotopes lines (green and red line).

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**Acknowledgment:** The work was partially support from the National Science Centre (DEC-2014/14/M/ST7/00866) and from the Statutory Found of the Chair of Electromagnetic Field Theory, Electronic Circuits and Optoelectronics (Faculty of Electronics, Wroclaw University of Science and Technology).

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# Detectors & Sources in MIR I

### Single-Mode Tunable VCSELs for the 2-4µm Wavelength Range

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Laser applications requiring single-mode, wavelength-tunable and/or low-power laser sources greatly benefit from using vertical-cavity surface-emitting lasers (VCSELs) instead of the more common edge-emitting semiconductor lasers. This particularly holds for tunable diode laser absorption spectroscopy (TDLAS), which is an import application field for nearand mid-infrared laser diodes [1]. Emitting in a single longitudinal mode with threshold currents of the order 1mA and high power-efficiency, near-infrared (up to ~1.3μm) VCSELs based on GaAs have conquered wide application areas ranging from high-bitrate datacom links to mass products such as laser mice [2, 3]. With successive improvements and advanced active region designs, their InP-based counterparts meanwhile cover the entire wavelength range from 1.3μm to about 2.6μm [4, 5]. These devices operate in a single transverse and longitudinal mode and can continuously be tuned without mode jumps over ~ 10 nm and ~ 100 nm by electrothermal and micromechanical tuning, respectively [6, 7]. Further extension into the mid-infrared has been achieved with GaSb-based VCSELs that today offer single-mode and tunable laser operation in the 2.3-4.0µm wavelength range [8, 9]. Experimental demonstrations of successful TDLAS applications have clearly proven the suitability of VCSELs, particularly in low-power and mobile systems [10].

We present advanced material concepts and quantum well designs to improve the long-wavelength performance of InP- and GaSb-based lasers and VCSELs. We describe in detail the device concept of the buried-tunnel-junction (BTJ-) VCSEL that revealed most successful for InP- and GaSb-based devices enabling high-performance continuous-wave lasing. Typically, the GaSb-based BTJ-VCSELs show polarization-stable single-mode lasing with threshold currents in the milliamp range and continuous electrothermal tuning ranges exceeding 10 nm.

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### INVITED

# Recent Progress in Development of Mid-IR Detection Modules for Gas Analyzers

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This year marks the 30th anniversary of the creation of Vigo System S.A., a company whose mission is to develop and manufacture high quality mid-infrared photodetectors operating at near-ambient temperatures (the "HOT" detectors). Cooling is an efficient way to improve performance of photodetectors. However, the use of heavy and costly cryogenic coolers is a barrier to the more wide spread use of infrared technology. The Vigo goal is to achieve sensitivity of the HOT devices close to the fundamental limits and GHz range frequency response. Our solution is the integration of optical, detection and electrical functions, such as concentration of incoming radiation, enhanced absorption, suppression of noisy thermal generation in a monolithic heterostructural chip [1,2]. The practical IR devices were initially based on Isothermal Vapor Phase Epitaxy, replaced in 2003 by Metal-Organic Chemical Vapor Phase Epitaxy of complex HgCdTe heterostructures. Recently, Molecular Beam Epitaxy grown InAsSb heterostructures have been also used.

The photocurrents generated by the HOT devices are usually small so low noise preamplifiers and signal processing systems are required to achieve the potential performance in practice

This paper reports on recent progress in infrared detection modules that integrate infrared photodetectors, optics, analog and digital signal processing, coolers, heat dissipation systems and other components in common packages. The integration makes detectors less vulnerable to electromagnetic interferences, overbias, electrostatic discharges, and other environmental exposures. The additional advantages of integration are improved HF performance, output signal standardization, miniaturization and cost reduction. This also significantly increases time-to-market for sensor systems. The high sensitivity and fast response devices can be optimized for any wavelength within the 3 to 16  $\mu$ m spectral range as required for a large variety of optoelectronic chemical sensors.

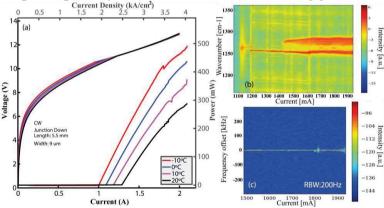
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# Stable, High-Power Quantum Cascade Laser Frequency Combs Operating in Room Temperature

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Quantum cascade lasers (QCLs) are compact and powerful semiconductor light sources, based on intersubband transitions, which can be tailored to operate in the mid-infrared (MIR) region of the electromagnetic spectrum [1]. Recently, it was demonstrated that QCLs can also operate as frequency combs [2], that can be applied in various dual-comb spectroscopy schemes [3].

In this work, we demonstrate the operation of high power QCL frequency combs, fabricated following a buried heterostrucutre protocol [4]. The top cladding of the device, was optimized to reduce the group delay dispersion (GDD), making use of a method for GDD waveguide engineering with a plasmon-fundamental mode resonance [5].



**Fig. 1** (a) Light-Current-Voltage (LIV) curves of a 5.5mm long and 9μm wide QCL comb (b) Spectrum heat map as a function of bias current. (c) Heat map of the Radio-Frequency (RF) beatnote. The FWHM of the beatnote is about 1kHz. Resolution bandwidth was 200Hz.

In figure 1, the LIV curves show continuous wave operation with an optical output of up to 300 mW at 20 °C. Comb operation starts at about 1.2 A, with a massive spectrum broadening to 60 cm<sup>-1</sup> at about 1.4 A. The comb remains stable up to 2 A, as shown by the narrow FWHM of the beatnote, measured with a resolution bandwidth of 200 Hz, in figure 1(c).

In conclusion, we fabricated and showed the operation of high power and stable QCL frequency combs that cover a large operating window.

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### MoO7

# GaSb-based Resonant Tunneling Structures with Ternary Prewell Injectors for Room Temperature Mid-Infrared Applications

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The semiconductors of the so-called 6.1 Å family, GaSb, InAs and AlSb, enable a wide range of bandgap energies and bandgap alignments, which makes them particularly suitable for mid-infrared optoelectronic semiconductor devices. Resonant tunneling diode (RTD) photodetectors based on 6.1 Å family semiconductors allow to transfer the photodetection principle of RTDs from the visible and near infrared to the mid-infrared wavelength region. The RTD photodetection principle originates from the modulation of the majority carrier resonant tunneling current via Coulomb interaction of photogenerated minority charge carriers. The RTD photodetection principle benefits from a low voltage operation, high amplification factors, low noise operation, and an increased functionality due to the RTD's distinct region of negative differential conductance. Here, we show room temperature operation of antimonide-based resonant tunneling diodes with GaSb/AlAsSb double barrier structures and pseudomorphically grown prewell emitter structures comprising the ternary compound semiconductors GaInSb and GaAsSb [1]. The AlSb/GaSb double barrier RTS resembles conventional AlGaAs/GaAs RTDs and provides a well-defined type-I band alignment with large offsets in conduction and valence band. We show that the electronic transport properties of AlSb/GaSb RTDs emitter prewells with large peak-to-valley current ratios are due to the increased  $\Gamma$ -L-valley energy separation and consequently an increased population of electrons at the  $\Gamma$ -point. At room temperature, resonant tunneling is absent for diode structures without prewell emitters but reaches 1.45 and 1.36 for samples with incorporation of Ga<sub>0.84</sub>In<sub>0.16</sub>Sb and GaAs<sub>0.05</sub>Sb<sub>0.95</sub> prewell emitters. RTD photodiodes with cut-off wavelength up to 3.5 µm are realized by integration of a lattice-matched GaInAsSb absorption layer. Furthermore, our findings have potential impact on alternative optoelectronic semiconductor devices of the mid infrared spectral region that exploit resonant tunneling transport mechanisms such as interband cascade lasers, interband cascade detectors and quantum cascade lasers.

[1] Andreas Pfenning; Georg Knebl; Fabian Hartmann; Robert Weih; Andreas Bader; Monika Emmerling; Martin Kamp; Sven Höfling; Lukas Worschech; *Appl. Phys. Lett.* **2017**, 110, DOI: 10.1063/1.4973894

### Analysis of Heat Dissipation Schemes in InGaAs/AllnAs/InP QCLs

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Thermal optimization of QCLs allows reaching higher output powers as well as more reliable devices. In this paper, we present detailed experimental investigation of thermal characteristics of the lasers based on CCD thermoreflectance (CCD TR). This method enables rapid thermal characterization of QCLs, as the registration of high-resolution temperature distribution map of the whole device takes only several seconds.

Experimental results are followed by numerical simulation of heat dissipation in QCLs. FEM analysis is performed for different QCL geometries. Good agreement of calculations with experimental data was obtained. Based on developed numerical model, improvements of device design leading to better overall thermal performance of QCLs are discussed.

All devices investigated were developed at the Institute of Electron Technology in cooperation with Faculty of Microsystem Electronics and Photonics, Wroclaw University of Technology. Devices concerned in this work are InGaAs/AlInAs/InP QCLs fabricated by either MBE technology or by hybrid MBE+MOVPE technology. Different architectures of devices have been compared in terms of heat management. Fig. 1 presents exemplary QCL CCD TR temperature distribution map for epi-down mounted buried laser together with calculated temperature distribution. The temperature increase is referred to the heat sink temperature. We have observed improvement of thermal properties of devices based on InP materials comparing to GaAs based devices [1]. From among all studied devices, i.e., single mesa, double trench and buried heterostructure (BH), the last ones showed the best thermal properties. The further reduction of heat dissipation is expected by optimizing device internal design and lowering threshold voltage.

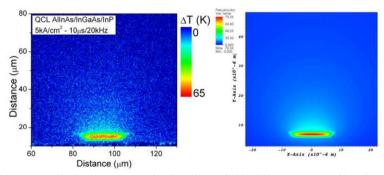


Fig. 1. Temperature distribution maps on the front facet of buried heterostructure AlInAs/InGaAs/InP QCL, experimental (left) and numerical (right). Devices were mounted epi-down.

This work was partially supported by National Science Centre grant OPUS 9 2015/17/B/ST7/04015.

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### MoO9

# Type-II Superlattice Photodetector Developments in the Mid-Infrared Region

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High-performance infrared photodetectors are currently of strong interest as successful building blocks for laser-based optical spectroscopic sensors as well as in imaging and threat warning systems. Here, we focus on Antimony-based type-II superlattice (T2SL) photodetectors, considered as the III/V-alternative to HgCdTe, for the infrared spectral range between  $3-12\mu m$ .

While the introduction of different heterojunction concepts has led to an enormous reduction of the generation recombination dark current due to the suppression of Shockley–Read–Hall processes in sophisticated mid-wave detector arrays, this allows nowadays to concentrate on operation at increased temperature without any loss in performance.

A new task also comprises to merge III-V and silicium technologies to include high-performance single-element photodetectors on uncooled photonic integrated circuits for applications in chemical sensing and Raman spectrometers. At present, thanks to a profoundly flexible fabrication technology, we develop photoconductive as well as photovoltaic detection devices for a broad spectral range within 3-12µm at high-operating temperatures with different shapes and sizes. To design this novel generation of uncooled or, at least just thermoelectric cooled InAs/GaSb detectors, we have implemented the four-component superlattice empirical pseudopotential method (SEPM) at room temperature (300K). This technique [1] includes both interface layers between InAs and GaSb as well as the material composition of the individual layers. It turned out, that the incorporation of the As-content in the barrier and interface material is a crucial parameter in our performed simulations which help to tailor appropriate bandgap-engineered devices. First results at these high-operating temperatures will be demonstrated and discussed.

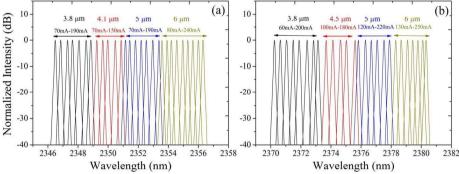
The recent investigations at Fraunhofer IAF in the field of InAs/GaSb T2SL technology [2] will therefore pave the road to future designs of single-element detectors: In cooperation with our European industrial partner micro-lenses shaped into the substrate beneath the detector element will account for increasing the signal-to-noise ratio. Via an international pilot line, executed among different research institutions and laboratories, these detectors will finally be integrated with thermoelectric coolers in a detection module and thus secure a new level of improvement in single-element photodetector technology.

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# Widely Tunable 2.3 $\mu m$ InP-Based Type-II DFB Laser Array Heterogeneously Integrated on Silicon for Sensing

R. Wang<sup>1,2</sup>, S. Sprengel<sup>3</sup>, G. Boehm<sup>3</sup>, R. Baets<sup>1,2</sup>, M. -C. Amann<sup>3</sup>, G. Roelkens<sup>1,2</sup>

Recently developed silicon photonics platforms offer possibilities to realize miniature and low-cost optical gas sensors. Although the silicon photonics platform is well-developed for the telecommunication wavelength range, development of silicon photonics integrated circuits (PICs) in 2-3  $\mu$ m range can have a broad range of applications since most important industrial gases have strong absorption lines in this wavelength range. For an integrated gas sensor based on tunable diode laser absorption spectroscopy technology, a tunable single mode laser on silicon is the key component that should be developed. Here we demonstrate a widely tunable InP-based type-II DFB laser array heterogeneously integrated on silicon.



**Fig. 1.** Emission spectra of heterogeneously integrated DFB laser arrays with grating period of 353 nm (a) and 357 nm (b).

Detailed information about the InP type-II DFB laser device structure can be found in [1]. In continuous-wave (CW) regime, the laser can operate up to  $25\,^{\circ}$ C, achieve an output power of 3 mW and have a threshold current density of  $1.6~\text{kA/cm}^2$  at a heat-sink temperature of  $5\,^{\circ}$ C. The CW operated laser array covers a wavelength range from  $2.28~\mu m$  to  $2.43~\mu m$  as the DFB grating period (defined in the silicon waveguide layer) varies from 343 nm to 368nm. 1nm change in DFB grating period results in  $\sim 6~\text{nm}$  shift in lasing wavelength. In order to achieve continuous tuning in the laser array, lasers with different III-V waveguide width are fabricated. Figure 1 shows the normalized emission spectra of two DFB laser arrays with grating period of 353 nm and 357 nm respectively. Each array consists of four lasers with different III-V waveguide width. The spacing ( $\sim 2.5~\text{nm}$ ) of the lasers in the arrays is sufficiently small such that each array can continuously cover a 10 nm wavelength range by tuning the injected current. The DFB laser array can be used to simultaneously detect several gas species with a single III-V-on-silicon sensor.

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### **MoO11**

# Carrier Dynamics in GaSb- Based Quantum Wells Emitting in the 2µm Spectral Range

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There are several experimental approaches which allow to look for improvement of the active part of emitters based on semiconductor low-dimensional systems. It is worth noticing that such activities as optimization of confining potential of the applied quantum wells, detection of the possible carrier escaping channels or improvement of the overlap between electrons and holes wavefunctions are important from the point of view of quantum efficiency, gain broadening, and emission power and consumption of the final device. On the other hand, any modifications in the active part also cause changes in carrier dynamics within the modified low-dimensional system. Knowledge of carrier life times and their dynamic processes are crucial to determine characteristics of the pulsed lasers such as 'mode lock' devices. In this presentation we will focus on possible ways of experimental determination of information about carrier dynamics in the low-dimensional systems e.g. in Type I and type II quantum wells grown on GaSb substrate, predicted for emission light in the mid infrared range (2-8µm).

Due to lack of fast and friendly in use streak cameras, working well (with 10ps time resolution), in visible or near-infrared range, in mid-infrared range time domain spectroscopy requires much more attention. Possible measurements require fast detectors and strong detected signals in case of one channel detector based measurements, sophisticated adjustment and proper crystals to transverse signals from mid- to near-infrared in case of upconversion measurements, advanced beams manipulation in experiments with mixing of the wavelength of several lasers, and finally, expensive, broadly tunable pulsed lasers for pumpprobe measurements. In this presentation we will focus on a technique based on single photon detection realized by using highly sensitive superconducting detectors. The results obtained for quantum wells emitting close to 2µm allowed to determine typical characteristic life times for confined carriers. One nanosecond life time was determined for type I InGaSb/GaSb quantum wells emitting in 1.65-1.8 µm spectral range, and almost two time longer (2ns) for type II GaSb/AlSb/InAs/GaInSb/AlSb/GaSb emitting at 1.8 µm. In addition, spectra obtained at different temperatures and at varied excitation power densities will be discussed. Finally, we hope that such an experimental approach will allow to extend possibility of its realization in the broader spectral range up to 10µm.

The work has partly been realized within iCspec project which received funding from the European Commission's Horizon 2020 Research and Innovation Programme under grant agreement No. 636930, and has also been supported by the National Science Centre of Poland within Grant No. 2014/15/B/ST7/04663.

## 16 May (Tuesday)

### Sessions

Laser sources I Laser sources II Poster Session Sensing systems III



Wrocław University of Science and Technology Main Building

### Strain-compensated AllnAs/InGaAs/InP quantum cascade lasers

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In this paper we report on fabrication of InP-based strain-compensated mid-IR QCLs designed for the emission at 4.7 µm. Three types of structures (see Fig.1) were investigated; the one grown exclusively by MBE without MOVPE overgrowth (structure A), the second fabricated by using hybrid approach combining MBE grown AlInAs/InGaAs active region with MOVPE grown InP waveguide layer (structure B) and the third one grown by MOVPE in full (structure C). Growth of the active region was performed by solid source MBE on Riber Compact 21T reactor. Epitaxial overgrowth (Structure B) and structure C were performed using AIXTRON 3x2" LP-MOVPE reactor.

500nm	InGaAs	n=8e18cm <sup>-3</sup>	Upper	
2.5µm	InAlAs	n=1e17cm-3	Waveguide	
500nm	InGaAs	n=4e16cm <sup>-3</sup>	1	
			Active Region	
500nm	InGaAs	n=4e16cm <sup>-3</sup>	Lower	
500µm Substrat	InP	n=2e17cm <sup>-3</sup>	Waveguid	

500nm	InP	n=8e18cm <sup>-3</sup>	Upper
1.5µm	InP	n=1e17cm <sup>-3</sup>	(MOVPE)
1.5µm	InP	n=4e16cm <sup>-3</sup>	
500nm	InGaAs	n=4e16cm <sup>-3</sup>	
			Active Region
500nm	InGaAs	n=4e16cm <sup>-3</sup>	Lower
500um Substrat	InP	n=2e17cm <sup>-3</sup>	waveguide

500nm	InP	n=8e118cm <sup>-3</sup>	Upper	
1,5um	InP	n=1e17cm <sup>-3</sup>	Waveguide	
1.5um	InP	n=4e16cm <sup>3</sup>		
			Active Region	
1,5um	InP	n=4e16cm <sup>-3</sup>	Lower	
1,5um	InP	n=1e17cm <sup>-3</sup>	waveguide	
500um Substrat	InP	n=2e18cm <sup>-3</sup>		

Fig.1a The layer sequence (structure A) Fig.1b The layer sequence (structure B) Fig.1c The layer sequence (structure C)

In the structure A the AlInAs/InGaAs active region of the laser, grown on lightly doped (n=2x10<sup>17</sup>cm<sup>-3</sup>) InP substrate serving as the bottom waveguide, was combined with 2.5 μm, low doped  $(n=1\times10^{17}\text{cm}^{-3})$  Al<sub>0.477</sub>In<sub>0.523</sub>As top waveguide layer and the heavily  $(n=8\times10^{18}\text{ cm}^{-3})$  doped In<sub>0.527</sub>Ga<sub>0.473</sub>As contact layer. The strain compensated Al<sub>0.638</sub>In<sub>0.362</sub>As/In<sub>0.669</sub>Ga<sub>0.331</sub>As/InP active core consisted of the 50-segments. The active region of the laser was of 4-well 2-phonon resonance design [1]. The layer sequence of one period of the structure, in nanometers, starting from the injection barrier was: 3.8, 1.2, 1.3, 4.3, 1.3, 3.8, 1.4, 3.6, 2.2, 2.8, 1.7, 2.5, 1.8, <u>2.2</u>, <u>1.9</u>, <u>2.1</u>, <u>2.1</u>, 2.0, **2.1**, 1.8, **2.7**, 1.8 nm [2]. The AllnAs layers are denoted in bold. The underlined layers are n doped to  $2.5 \times 10^{11}$  cm<sup>-2</sup>. The high resolution X-ray diffraction (HRXRD) (see Fig.2) shows that grown structure was almost exactly as designed. The lattice mismatch of the whole structure to the InP substrate was as low as 250 ppm.

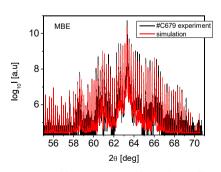


Fig.2 XRD-2 $\Theta/\omega$  scans measured for InP (004) diffraction condition of the QCL epitaxial structure grown by MBE (structure B).

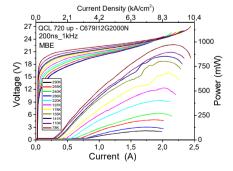


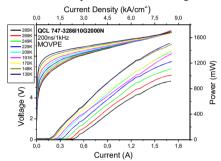
Fig.3 L-I-V characteristics of the Al<sub>0.638</sub>In<sub>0.362</sub>As/In<sub>0.669</sub>Ga<sub>0.331</sub> As/InP laser ( $\lambda$ =4.4  $\mu$ m) grown by MBE (structure A).

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The double trench lasers were fabricated using standard processing technology, i.e., wet etching and  $Si_3N_4$  for electrical insulation. The lasers were cleaved into bars of 2 mm long and soldered episide up to Au-plated AlN submounts. The lasers emitted at 4.4  $\mu m$  at 300K. The light–current and current-voltage characteristics of the 12  $\mu m$ -laser are shown in Fig.3. The threshold current density at 77 K was 1.15 kA/cm² and raised to  $\sim 3.25$  kA/cm² at 300 K.

In the structure B the AlInAs/InGaAs active region of the laser was grown by MBE on lightly doped (n= $2x10^{17}$ cm<sup>-3</sup>) InP substrate serving as lower waveguide. Than InP top waveguide with graded doping (1.5  $\mu$ m, n =  $4x10^{16}$  cm<sup>-3</sup> followed by 1.5  $\mu$ m, n =  $1x10^{17}$  cm<sup>-3</sup>) and contact layer (n =  $8x10^{18}$  cm<sup>-3</sup>) were grown by MOVPE. The design and doping of the active region was the same as before. Applying the same processing we have got lasers with 2.6 kA/cm<sup>2</sup> threshold current density at 300 K.

The structure C, grown by MOVPE, had the same design, except for that active core consisted of 30-segments instead of the 50. After standard processing the uncoated lasers showed 1.25 kA/cm<sup>2</sup> threshold current density at 77 K and 2.18 kA/cm<sup>2</sup> at 300 K (see Fig.4). The lasers showed multilongitudinal mode behavior with emission spectrum envelope peaked at  $\lambda$ =4.75  $\mu$ m (see Fig.5). The characteristic temperature for MOVPE grown lasers was  $T_0$ =155 K.



Wavelength (μm) 4,88 4.76 4,65 4,55 0,54A 0,644 0.824 1.164 ntensity (a.u.) 1.37A QCL 747-3286I10G2000 96K/200ns/5kHz 2000 2050 2100 2150 Wavenumber (cm<sup>-1</sup>)

Fig.4 Light–current and current-voltage characteristics of the  $Al_{0.638}In_{0.362}As/In_{0.669}Ga_{0.331}$  As/InP laser ( $\lambda$ =4.4  $\mu$ m) grown by MOVPE (structure C).

Fig.5 Emission spectra of the Al<sub>0.638</sub>In<sub>0.362</sub>As/In<sub>0.669</sub>Ga<sub>0.331</sub> As/InP laser (λ=4.75 μm) grown by MOVPE (structure C) at 300 K.

The investigated lasers have been analyzed with the aim of non-equilibrium Green's function (NEGF) method demonstrating capability of this approach in predicting basic operational characteristics of the devices [3,4]. The calculated threshold current densities are in well agreement with experimental values.

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### **Recent Progress in Interband Cascade Devices**

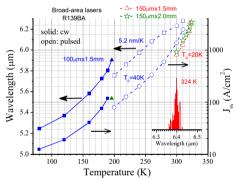
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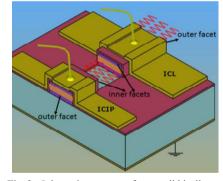
The family of interband cascade (IC) devices includes lasers [1-3], infrared photodetectors [4] and photovoltaic cells [5]. These devices take advantage of the broken bandgap alignment in type-II quantum wells to realize cascade stages for high device performance.

We present our recent progress in IC devices including: InAs-based IC lasers [6], long-wavelength IC infrared photodetectors [7], and a monolithically integrated IC laser and photodetector [8]. By employing an improved waveguide structure, the 18-stage InAs-based IC lasers lased above room temperature at wavelengths beyond 6  $\mu$ m (see Fig. 1). The threshold current density was as low as 810 A/cm² at 300 K, although the material quality was not optimal. A monolithically integrated mid-IR type-I IC laser and photodetector was demonstrated at room temperature (see Fig. 2). The detectivity of the uncooled detector section is as high as  $1.9\times10^{10}$  Jones. Updated results on our research and development of IC optoelectronic devices will be presented at the workshop.

This work was partially supported by the NSF (ECCS-1202318 and IIP-1346307) and AFSOR (FA9550-15-1-0067).



**Fig. 1.** Threshold current density ( $J_{th}$ ) and lasing wavelength. The inset is the lasing spectrum at 324 K.



**Fig. 2.** Schematic structure of a monolithically integrated mid-IR IC laser and photodetector

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### TuO<sub>1</sub>

# Fully-fiberized Multi-wavelength Difference Frequency Generation Mid-infrared Source for Laser Spectroscopy Applications

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Optical-based trace gas detection techniques are being implemented in various applications: homeland security, medicine, industry, environmental monitoring and several more [1]. Some sophisticated applications require simultaneous concentration monitoring of several gas molecules. As the strongest absorption lines often lie in the mid-IR wavelength region this requires using independent laser sources, each targeting a separate absorption profile [2]. We present a fully-monolithic, fiber-based difference frequency generation (DFG) mid-IR source, incorporating our patented dual-wavelength double-clad (DC) configuration, which enables amplification of  $\sim 1.55~\mu m$  radiation and simultaneous generation of 1.064  $\mu m$  wavelength [3], which are required in the DFG process. Schematic is shown in Fig. 1.

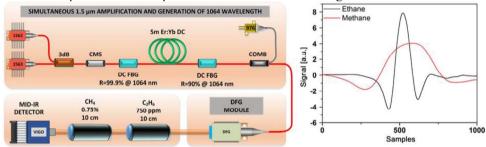


Fig. 1 System setup (left) and 2f absorption signals registered simultaneously for ethane and methane gas cells (right). 3db – fiber coupler, CMS – cladding mode stripper, DC FBG – double clad fiber Bragg grating, COMB –combiner, DFG – difference frequency generation, Er:Yb DC – double clad fiber doped with erbium and ytterbium.

The proposed mid-IR source is built in two main parts. The first part consists of a Er:Yb DC fiber amplifier which amplifies emission of low power seed diodes (~1562 and 1563 nm) and simultaneously generates 1064 nm radiation via the patented dual-wavelength configuration. The amplified 1.56  $\mu m$  and generated 1.064  $\mu m$  radiation is delivered to a fiber-pigtailed difference frequency generation (DFG) PPLN module (NEL), via a single-mode fiber. The generated Mid-IR beams were used to instantaneously detect two independent absorption lines of gas species, enclosed in 10 cm long absorption cells: methane (at 760 Torr) and ethane (at 200 Torr), using WMS technique. In order to enable simultaneous analysis of two separate absorption lines with a single mid-IR detector, we used dissimilar sine-wave modulation frequencies for each seed laser, therefore permitting detection of 2f signals from the ethane and methane absorption lines separately. The proposed setup is modular and supports non-complex replacement of the 1.5  $\mu m$  seed diodes, which in turn enables tailoring the mid-IR emission to reach any gas absorption line in the range of 2930 – 3010 cm $^{-1}$ .

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### Acknowledgments

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# Dual-wavelength DFB Quantum Cascade Lasers for NO and NO<sub>2</sub> Sensing

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The Quantum Cascade Laser (QCL) is a unipolar laser based on intersubband transitions in a semiconductor heterostructure, which can be tailored to operate in the mid-infrared (MIR) region of spectrum [1]. This region is of particular interest for gas absorption spectroscopy [2]. Simultaneous detection of multiple gases requires light sources capable of emitting at several wavelengths [3]. Here, two novel designs of dual-wavelength devices based on distributed feedback (DFB) grating QCLs fabricated next to each other and dual section lasers based on Vernier effect will be presented.

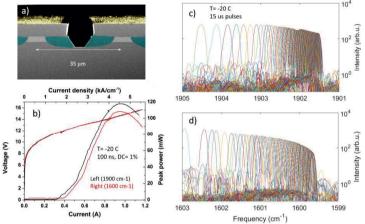


Fig. 1 a) SEM image of neighbor DFB QCLs, b) LIV characteristics and c,d) spectral evolution analysis of 3mm long and 7  $\mu$ m wide lasers with ridge to ridge distance of 35  $\mu$ m.

The fabrication process is based on an inverted buried heterostructure protocol [4], with active regions consisting of a double heterogeneous quantum cascade stack of InGaAs and InAlAs layers. Fig. 1 shows the scanning electron microscopy (SEM) image as well as the light-current-voltage (LIV) and spectral evolution analysis of 3 mm long and 7  $\mu$ m wide neighbor DFB QCLs at T= -20 °C. Each laser remains single mode, while the frequency of each laser is continuously tuned around the target absorption frequencies. The separation distance of lasers was chosen to be 35 $\mu$ m in order to have electrical isolation with a wet-etching process and still allowing single-beam collimation. The active region and waveguide of lasers were designed in order to have low and comparable lasing thresholds, being 320mA (1.5 kA/cm²) and 380mA (1.9 kA/cm²) at 1900 cm¹ and 1600 cm¹. Using these DFB-QCLs in a spectroscopic setup, combined with a 36m long-path gas cell, the concentration of NO and NO<sub>2</sub> gases was simultaneously measured, achieving a high precision of 0.2 ppb and 0.06 ppb for 200 s acquisition time. The results of another design of dual wavelength QCLs based on Vernier effect with integrated heaters on the side of laser allowing for switching of the frequency between 1600 cm¹ and 1900 cm¹ will be presented as well.

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Nano-Tera.ch is acknowledged for financial support of this work.

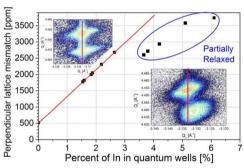
### TuO3

### MBE Growth and Fabrication of In<sub>x</sub>Ga<sub>1-x</sub>As/Al<sub>0.45</sub>Ga<sub>0.55</sub>As/GaAs Strained Quantum Cascade Lasers

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In this work we present Molecular Beam Epitaxy (MBE) growth of strained  $In_xGa_{1.x}As/Al_{0.45}Ga_{0.55}As/GaAs$  Quantum Cascade Lasers (QCLs). Modification of the active region superlattice based on replacing the GaAs quantum wells by InGaAs with few percent of indium was applied. The structures have been studied by high resolution X-ray diffraction technique. The structural characterization of samples shows that there is linear dependence between the perpendicular lattice mismatch and indium content less than 3%. For samples with greater indium stoichiometric coefficient the linear dependence is not observed – structures are partially relaxed. Devices fabricated from strained heterostructures containing up to 3% of indium exhibited performance largely improved over  $Al_{0.45}Ga_{0.55}As/GaAs$  QCLs (Fig.2). The In containing QCLs outperform standard  $Al_{0.45}Ga_{0.55}As/GaAs$  lasers in terms of threshold current and maximum operating temperature [1].



77 K QCL GAASA/GAAS; — 250 K QCL GAASA/GAAS 77 K QCL InGAASA/GAAS; — 250 K QCL InGAASA/GAAS 77 K QCL InGAASA/GAAS; — 250 K QCL InGAASA/GAAS 8 77 K QCL InGAASA/GAAS; — 250 K QCL InGAASA/GAAS 8 7 6 8 10 12 14 12 14 10 12 14 12 14 10 12 14 10 12 14 10 12 14 10 12 14 10 12 14 10 12 14 10 12

**Fig.1.** Superlattice mismatch as a function of indium content in  $In_xGa_{1-x}As/Al_{0.45}Ga_{0.55}As/GaAs$  QCLs is presented. The asymmetric reciprocal space maps are shown.

**Fig.1.** The electro-optical characteristics of reference Al<sub>0.45</sub>Ga<sub>0.55</sub>As/GaAs QCL structure (black), In<sub>0.026</sub>Ga<sub>0.974</sub>As/Al<sub>0.45</sub>Ga<sub>0.55</sub>As/GaAs QCL (red) and partially relaxed In<sub>0.051</sub>Ga<sub>0.949</sub>As/Al<sub>0.45</sub>Ga<sub>0.55</sub>As/GaAs (blue)

The lasing was achieved in pulsed mode up to  $T=50^{\circ}\mathrm{C}$  with characteristic temperature  $T_0=120~\mathrm{K}$ . The values of  $2.2~\mathrm{kA/cm^2}$  at  $77~\mathrm{K}$  and  $7.0~\mathrm{kA/cm^2}$  at  $250~\mathrm{K}$  are the lowest threshold current densities ever obtained for GaAs based quantum cascade lasers emitting in  $\sim 9~\mu m$  range. The emitted power is also considerably higher in the case of  $In_xGa_{1.x}As/Al_{0.45}Ga_{0.55}As/GaAs$  QCL, especially at elevated temperatures. At  $300~\mathrm{K}$   $In_{0.026}Ga_{0.974}As/Al_{0.45}Ga_{0.55}As/GaAs$  QCL emit the maximal peak power  $\sim 0.5~\mathrm{W}$ . Devices fabricated from  $In_{0.051}Ga_{0.949}As/Al_{0.45}Ga_{0.55}As/GaAs$  partially relaxed heterostructure showed gradually degradation of the performance. Reduction of the threshold current density in strained QCLs is attributed to the decrease of thermal carrier escape from the upper laser level and reduction of interface roughness scattering. This work shows the potential of GaAs based QCLs to become useful sources of mid-infrared radiation for specific applications.

This work was partially supported by NCN grant PRELUDIUM 9, 2015/17/N/ST7/03936 (IBIS).

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### Mid-Infrared Supercontinuum - A Maturing Technology

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It has been over a decade since the generation of ultrabroadband spectra know as supercontinua were first demonstrated in the mid-IR using soft glass fibers [1] and the widespread application and commercial breakthrough of this technology has not appeared due to the challenges in making reliable commercially mature sources. However, recently the availability of these sources has grown dramatically with half a dozen suppliers now offering sources commercially.

In this talk NKT Photonics, the world's leading manufacturer of visible supercontinuum sources and one of the earliest parties to begin the development of mid-IR supercontinuum sources will talk about the challenges involved in producing mid-Infrared supercontinuum sources and demonstrate that they have been overcome and that supercontinuum sources with many thousands of hours reliability have been built. We will touch upon the future directions in which mid-IR supercontinuum



**Figure 1:** Fully packaged, high repetition rate turnkey mid-IR supercontinuum system from NKT Photonics.

is presently developing, such as the application of new materials [2] and fiber cascades [3]. Finally we will describe the potential for highly flexible supercontinuum gas sensing that we see.

The field of fluorescence microscopy has over the last decade been revolutionized by the implementation visible supercontinuum systems. Now the emergence of commercial supercontinuum sources in the mid-IR could herald a similar revolution in mid-IR sensing, spectroscopy and microscopy[4-6].

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#### TuO<sub>5</sub>

#### **Quantum-Cascade Vertical-Cavity Surface-Emitting Laser**

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This paper proposes a design for the first quantum-cascade vertical-cavity surface-emitting laser (QC VCSEL). Presently QC lasers can be fabricated in horizontal cavity geometry since stimulated emission from quantum cascades is possible if electrical component of the electromagnetic wave is perpendicular to the QC surface. That field component is absent in vertical cavity, which makes fundamentally impossible fabrication of QC VCSELs in their conventional design.

In our novel design, the top VCSEL mirror is a monolithic high-refractive-index contrast grating (MHCG), which serves as both an optical coupler and as the region in which the vertical component of the electrical field is induced, enabling stimulating emission from the quantum cascades.

Using a three-dimensional, fully vectorial optical model, the properties of different MHCGs as possible QC VCSEL mirrors are analysed. The distribution of the optical field and the threshold gains of VCSELs with QCs embedded in MHCGs are also simulated.

Our analysis shows that the vertical component of the electric field of the electromagnetic wave can be induced in the region of the MHCG, where it stimulates emission in the quantum cascades embedded in the MHCG stripes and enables to reach the threshold for vertical laser emission.

The possibility of fabricating QC VCSELs opens perspectives for merging the advantages of VCSELs with those of QC lasers. QC VCSELs are expected to emit a good quality beam with minimal divergence, exhibit low threshold current, enable integration into two-dimensional arrays (as with VCSELs) and emit in the micrometer spectral range (as QC lasers do).

#### Electronic Band Structure and Material Gain in Gasb-Based Quantum Wells Containing Bismuth: Toward Enhancement of Quantum Confinement in the Valence Band

#### M. Gładysiewicz, M. Wartak and R. Kudrawiec

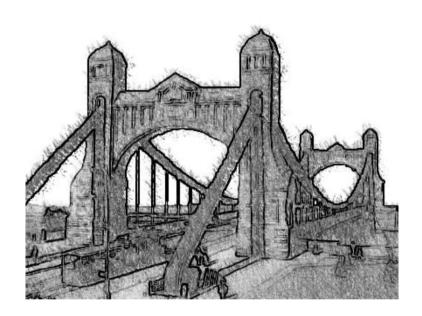
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The electronic band structure and material gain for compressively strained 8 nm wide III-V-Bi quantum wells (QWs) grown on GaSb substrates have been calculated using 8-band kp Hamiltonian [1, 2]. Bi-related changes in the electronic band structure of III-V-Bi alloys have been modeled according to ab-initio calculations for ternary alloys (III-Ga-Bi) [3]. It is shown that the gain peak strongly shifts to longer wavelengths due to Bi incorporation. For GaSb substrate we analyzed three Bi-containing OWs (GaSbBi, GaInSbBi, GaInAsSbBi) and different Bi-free barriers (GaSb and AlGaInAsSb) lattice matched to GaSb. When compared to Bi-free QWs the analyzed Bi-containing structures show much better quantum confinement in the valence band and also larger redshift of material gain peak per percent of compressive strain. For GaInSb/GaSb QWs, the gain peak is predicted at 2.1 µm for the compressive strain of ε=2% (32% In). The gain peak of GaSbBi/GaSb OW reaches this wavelength for compressive strain of 0.15% that corresponds to about 5% Bi. It has also been shown that replacing In atoms by Bi atoms in GaInSbBi/GaSb QWs while keeping the same compressive strain (ε=2%) in OW region, enhances and shifts gain peak significantly to the longer wavelengths, see Fig. 3. For GaInSbBi/GaSb QW with 5% Bi, the gain peak is predicted at around 2.6 µm i.e., is redshifted by about 400 nm compared to Bi-free QW. For 8 nm wide GaInAsSbSb OWs (80% In, 5% Bi and ε=2%) with proper AlGaInAsSb barriers it is possible to achieve large material gain even at 4.0 µm [2].

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#### Acknowledgements

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Grunwaldzki Bridge

## Calibration-free Wavelength Modulation Spectroscopy for Gas Sensing with a DFB QCL

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In this paper, we described the calibration-free wavelength modulation spectroscopy for practical gas sensing. The gas concentration can be calculated from the integrate absorption which retrieved by fitting the entire simulated 1f normalized 2f signal to the measured one. The simulation was based on the frequency response and the measured laser intensity. For time-varying laser frequency, it was modeled under an ideal assumption that the laser frequency response of the scan and modulation current is independent of each other. A homemade etalon was used to get the frequency response of the scan and modulation current respectively. And the position of the absorption transition was used to calibrate the absolute wavelength of the laser. For laser intensity, it was modeled by the measured laser intensity. In this way, all the intensity information was contained in the simulation, avoiding the need for numerical intensity models. The WMS harmonics of the simulation and the measured WMS absorption signal were extracted by the lock-in and filters, which avoided the need for their Fourier expansion. The measured 1f normalized 2f signal can be treat as a function of the integrate absorption and the absorption line-shape. In the simulation process, the absorption line-shape was described by Voigt absorption model which is the convolution of Doppler and collisional broadenings. The fitting routine was analogous to the technique used in direct absorption spectra fittings. The simulated 1f normalized 2f spectra was least-squares fit to the measured one with several free parameters (eg: center transition frequency v0, integrate absorption A and collision-broadened full-with at half-maximum  $\Delta v_1$ ). The best-fit parameters can be used to calculate gas properties as done in direction absorption technique. This calibration-free method is valid with any optical depth and modulation index.

A distributed feedback quantum cascade laser (DFB QCL) operating around 2055 cm<sup>-1</sup> was used and the absorption transition line of  $CO_2$  at 2055.16 cm<sup>-1</sup> was selected for the measurements. During the experiment, the scan range of the QCL is about 0.6 cm<sup>-1</sup> (2054.89  $\sim$  2055.42 cm<sup>-1</sup>) with the scan current (200 Hz, 145 mA  $\sim$  170 mA) and the modulation current (20kHz, 7mA). Different mixing ratio of  $CO_2$  (15%, 20%, 25%, 30%, 35%, 40%, 45% was flowed into a gas cell with an optical path of 25cm and the integrate absorption range is 1.9%  $\sim$  5.7%. During the experiment, the temperature of the gas cell is about 285K, and the pressure is 1atm.

The experiment results showed that the simulation and the measurements are in good agreement over the entire WMS line shape. The residual, define as the difference between the simulation and the measurements, is relative small. And the ratio of the residual to the corresponding peak value of the measurements is within 4%. There is a good linearity between the integrate absorption retrieved from the best-fit result and the known gas concentration (the linear fit coefficient > 0.998). The concentration was calculated from the integrate absorption with the parameters in HITRAN database. The measured error of the concentration is smaller than 4% (the smallest error is about 0.57%).

#### Acknowledgement

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#### Temperature-Resolved Photoluminescence and Photoreflectance Studies of GaSb-Based Resonant Tunneling Structures Operating in Mid-Infrared Spectral Range

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Resonant tunneling structures (RTS) play an important role in a large variety of nowadays electronic and optoelectronic semiconductor devices. Besides being applied in high-speed electronic circuits and even as THz emitters, resonant tunneling is the basic underlying physical principle used in various optoelectronic devices, such as quantum cascade lasers (QCL), interband cascade lasers (ICL) and interband cascade detectors (ICD). Compared with conventional RTSs based on the GaAs or InP material system as mostly employed in QCLs, the InAs-GaSb-AlSb material system offers a broad spectrum of bandgap energies and band alignments from staggered (type-I) to broken (type-II) [1] which e.g. enables their exploitation in light emitting and detecting optoelectronic devices covering the mid-infrared spectral range [2,3]. Substantial flexibility for band gap engineering offers the ability to design and study RTSs with various band alignments, i.e. AlSb/GaSb/AlSb, AlSb/InAs/AlSb. The exact energy structure of such complex systems is often not well known, due to for example interface layer formations.

In this work, we present a comprehensive investigation of the band structure of such a complex layered system. Methods of Fourier-transform spectroscopy were employed to investigate structures based on two asymmetric hybrid AlSb/InAs type-II barriers within a GaSb matrix. In this quantum system, the AlSb layer function as common type-I barrier for electrons and holes, whereas the InAs part functions a trap for electron confinement, while enhancing the hole barrier within the valence band. We find light emission in the mid-infrared spectral range between 3 and 8 µm. Formation of quasi-bound states in the region between the InAs/GaSb interface and the AlSb barriers was confirmed, allowing for resonant tunneling of carriers across the structure [4]. Since the structure under investigation contains two asymmetric type II quantum wells, two quasi-bound states were found.

This study was carried out with financial support from the National Science Center of Poland within Grant No. 2016/21/N/ST7/02790.

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#### Development of a Sensitive and Compact Nitric Oxide I-QEPAS Sensor System Based on a High-Finesse Cavity and 5.2 μm CW DFB QC

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Nitric oxide (NO) is known as toxic gas, the concentration of which is strongly related to meteorological conditions and emission sources (anthropogenic and natural). NO is also produced during combustion of fossil fuels in power plants, automobile engines and naturally e.g. during lightning in thunderstorms. NO can be observed in characteristic decomposition compounds of specific explosives materials [1]. Furthermore, NO plays important role at numerous functions in the human body where is produced in inflammatory processes [2].

We report an application of intracavity quartz-enhanced photoacoustic spectroscopy to midinfrared nitric oxide detection [3]. A detection limit of 4.8 ppbv within a 30 ms integration time was demonstrated for the first time by using a room-temperature, continuous-wave, distributed-feedback quantum cascade laser (QCL) emitting at 5.263 µm (1900.08 cm<sup>-1</sup>) and a new compact design of a high-finesse bow-tie optical cavity with an integrated resonant quartz tuning fork (QTF) (Fig.1). The optimum configuration of the bow-tie cavity was simulated using custom software. Measurements were performed with a wavelength modulation scheme (WM) using a 2f detection procedure. The QCL emission wavelength was matched to the molecular absorption spectrum of the nitric oxide based on QCL temperature and current measurements.

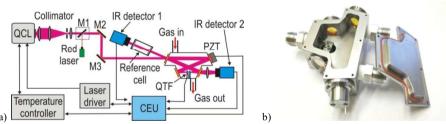


Fig. 1. a) Schematic of an I-QEPAS nitric oxide sensor platform, b) Novel and compact bow tie resonator for I-QEPAS sensor system.

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## Single-QCL-based Spectroscopy Absorption System for Simultaneous Measurements of CO and CO<sub>2</sub>

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We report the development of the absorption system for simultaneous detection of atmospheric CO and  $CO_2$  based on a single QCL, with a 74 m multi-pass cell. The two neighboring absorption lines of CO (2055.40 cm<sup>-1</sup>) and  $CO_2$  (2055.16 cm<sup>-1</sup>) are covered within a single wavelength scan. Wavelength modulation spectroscopy (f = 20 kHz) is utilized to enhance the signal-to-noise ratio. A relative lower pressure (250 torr) can be selected to avoid the spectral interference between  $CO_2$  and CO. The concentration calibration of the system was performed using standard gases with different concentrations, and the calibration results illustrated a rather good linear response for both CO (200 ppb–3.7 ppm) and  $CO_2$  (95 ppm–1001 ppm). A minimum detection limit of 1.3 ppb (1×10<sup>-9</sup>) for CO and 0.44ppm (1×10<sup>-6</sup>) for  $CO_2$  are achieved, by analyzing the Allan variance of the continuous detection results.

#### Acknowledgement

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#### Figures:

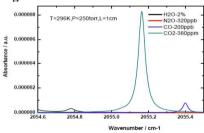
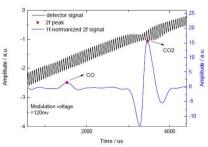
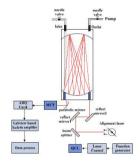


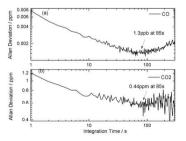
Fig. 1. The simulated absorption spectra for H<sub>2</sub>O, N<sub>2</sub>O, CO<sub>2</sub>, CO in air at 296K and 250torr based on HITRAN database



**Fig. 3.** An example of detector signal and related 2f signal of laboratory air



**Fig. 2.** Schematic of the experimental apparatus



**Fig. 4.** Allan Deviation plot of CO and CO2 as a function of integration time

## Fourier Transformed Photoluminescence Studies of GalnAsSb/AlGa(In)AsSb Type I QWs for 3-3.5 µm Emission

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Semiconductor light sources emitting in the mid-infrared spectral region (2–5 µm) are becoming increasingly appealing for gas sensing as well as medical and defense applications. In particular, gas sensing takes advantage of presence of numerous absorption lines for gases such as methane, ozone, carbon dioxide, and carbon monoxide. This has led to intensified research on the development of different semiconductor laser sources for this spectral range. There are several ways to obtain emission in this range considering different type of devices, such as laser diode [1](LD), Interband Cascade Lasers [2](ICL) and Quantum Cascade Lasers [3](QCL). All of them have independent advantages such as high output power in QCLs or high spectral tunability in ICLs. While laser diodes typically achieve high output powers and can exhibit single wavelength operation, the tunability of their spectra is rather small. In this contribution we will focus on GaSb-based type-I quantum wells as active parts of the superluminescence leds [4] (SLEDs) emitting in 2.65-3.5 µm wavelength range. The presented studies, based on temperature dependence and excitation power dependence of Fourier transformed photoluminescence measurements will focused on the modified GaInAsSb/AlGa(In)AsSb quantum wells aimed to provide emission wavelength up to 3.5 μm. With increasing indium composition in quantum wells emission wavelength was shifted to 2.5 μm (at 300K) and additional indium atoms application in barriers allowed for further shift up to 3 um. Nevertheless, the latter procedure results in additional peaks appearing in photoluminescence spectra, showing necessity of further growth optimization, from the point of view enhancing emission intensity and also extend the operation wavelength beyond 3 μm.

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## Type-I Direct Gap GeSn-Based Quantum Wells Integrated with Si Platform for Mid-Infrared Applications

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We have shown that GeSn-based quantum wells grown on a Ge substrate with various widths and Sn contents are very promising gain media for mid-infrared lasers which can be integrated with an Si platform. In our recent work, with reasonable α-Sn content and thickness ranges, we present compressively strained type-I single quantum well (I-QW) that can emit light in short wave part of mid-infrared spectrum region [1]. Because it is possible to close bandgap in Ge<sub>1-w</sub>Sn<sub>w</sub> alloys, this material is very perspective for emission with wavelength wider than in presented part of mid-infrared wavelength region. In order to show and analyze photon generation in proposed system, we have performed material optical gain spectra calculations, from which it is clearly seen that emission wavelength, can be tuned by thin film layer content and thickness [1]. Moreover it is obvious that polarization should be tunable so and controllable via strain incorporated to thin film. It can be achieved by smart changing content of virtual substrate, which in our proposition is a barrier material of QW. So, since in proposed structure there is Ge<sub>1-w</sub>Sn<sub>w</sub> as thin film and Ge as an unstrained barrier, transverse electric (TE) mode of optical gain is the one dominant in values while transverse magnetic (TM) mode of optical gain is negligible small in values [1]. However, TM mode gain can be achieved and even dominant one in Ge<sub>1-w</sub>Sn<sub>w</sub> QWs when there is compressive strain incorporated into thin layer. For example, wide strain engineering can be done via relaxed barriers made of ternary alloys Si<sub>v</sub>Ge<sub>1-x-v</sub>Sn<sub>x</sub> [2].

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## Type I and Type II Radiative Recombination an InAs-Based Narrow-Gap Heterostructures

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Semiconductor light sources emitting in the mid-infrared spectral region 3–3.5  $\mu$ m are becoming increasingly appealing for optical gas sensing market. In particular, detection of various hydrocarbons is possible due to their numerous absorption lines in this range. This has led to intensified research on development of different semiconductor laser sources with respect to lowering price of their fabrication processes. There are several ways to obtain emission in this range exploiting different types of devices, for example Interband Cascade Lasers (ICL) [1]. All of them have advantages such as high output power, high spectral tunability, single mode operation, etc. Nevertheless, most of the devices require high precision growth using Molecular Beam Epitaxy, which cause high production costs. That is a serious disadvantage in mass production of cheap lasing devices.

In this contribution we will focus on type I quantum wells, grown by cheaper technique i.e. metalorganic vapour phase epitaxy (MOCVD), which allows fabrication of InAsSbP epitaxial layers with ultimate phosphorus content isomorphic with the InAs matrix [2,3]. The InAsSbP/In(As,Sb) heterojuction exhibited type I and type II band alignment depending on the composition of the contacting materials. The presented studies, based on temperature dependence and excitation power dependence of Fourier transformed photoluminescence measurements [4] were focused on the InAsSbP/InAsSb/InAsSbP quantum wells, grown on the InAs substrate, and aimed to provide emission wavelength in range of hydrocarbon absorption (3.5 $\mu$ m at room temperature). In addition, structural properties of investigated structures will be discussed based on numbers of spectra, regarding performed X-ray and TEM measurements, in context of possibility of use investigated structures as an active region of the diode lasers.

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### Spatial Distribution of Stokes Parameters in Non-Gaussian Beam of Quantum Cascade Laser

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Quantum cascade lasers (QCLs) in principle emit light that is linearly polarized in the epitaxial growth direction, due to the polarization selection rule for intersubband transition [1]. However, measurement of Stokes parameters spatially averaged in QCL beam have shown presence of circularly polarized light [2].

In this paper we present experimental results of Stokes parameters spatial distribution in QCL beam. The results have been obtained by rotating quarter-waveplate measurements [3] in setup with wide-angle goniometric profiler [4]. The studied laser is mid-infrared straincompensated InAlAs/InGaAs/InP QCL grown by solid-source molecular beam epitaxy [5].

We observed rotation of the plane of polarization of light. The angle between the polarization plane and the epitaxial growth direction increases with distance from the center of the beam. The contribution of circularly polarized light is noticeable but small and almost constant.

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## **Characterization of Mid-Infrared Femtosecond Pulses by the Two- Photon Absorption Process and Up-Conversion Technique**

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Despite the rapid development of pulse laser sources operating in the mid-infrared (mid-IR) spectral range, their temporal characterization remains challenging. The standard characterization techniques based mostly on second harmonic generation (SHG) process in a nonlinear crystal is very sensitive and relatively easy applicable in a visible or near-infrared spectral range. However, these techniques are not easily transferred to the mid-IR due to a low efficiency of the SHG process at low peak power intensity and necessity to develop new nonlinear materials. Nevertheless, some interesting solutions can be considered as a reliable alternative to the standard techniques.

We present two time-domain characterization techniques of mid-IR, sub-ps and low peak power optical pulses employing optical intensity autocorrelation using either the two-photon absorption (TPA) or frequency mixing process via sum frequency generation (SFG) in a nonlinear crystal.

For the purpose of experiments the optical parametric oscillator (OPO) synchronously pumped by the titanium-sapphire laser has been used to generate a train of low peak power (pJ) sub-ps mid-IR pulses spectrally centered at ~3.5 μm. In the case of TPA technique, the output of the OPO is directed to a Michelson-type interferometer to produce two trains of pulses with a variable temporal delay settled with a 13 fs resolution. Subsequently, both pulse trains are focused on the commercially available InGaAs PIN diode for which the long wavelength limit of linear absorption reaches ~1.9 μm. As a result, we were able to obtain the photocurrent amplitude proportional to the non-linear TPA process coming from the interaction of two laser pulses as a function of temporal delay between them. A clear TPA response gave the function-corrected mid-IR pulse duration on the level of 250 fs. The origin of the non-linear TPA process is reflected in a quadratic response of the photocurrent with the incident laser power. In the case of the frequency mixing process, a specially designed periodically-pooled MgO crystal is employed able to produce the SFG product of two laser pulses at ~3.5 μm and 0.83 μm from the titanium-sapphire laser. Since the SFG is proportional to the intensity of interacting pulses thus gating in time of mid-infrared pulse by the infrared one within the crystal gives the temporal intensity profile of the SFG product. As expected, the duration of the mid-infrared pulse is similar to the one obtained by TPA technique and equals about 290 fs.

These two methods present possibilities for characterization of short sub-ps and low peak power pulses via intensity autocorrelation measurement. Especially, the TPA technique seems to be very promising. It requires a relatively chip, commercially available and handles photodiode, the experiment is very robust without a necessity to use any expensive polarizers desired for the mid-IR spectral range, and there is no particular geometrical configuration of laser beams.

The work has been realized within iCspec project which received funding from the European Commission's Horizon 2020 Research and Innovation Programme under grant agreement No. 636930.

## Carrier Dynamics in a W-type Quantum Well Emitting in the Mid-infrared Spectral Range Probed by the Transient Reflectivity Technique

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The type II W-design quantum wells (QW) based on InAs/GaInSb broken gap materials' combination have been developed as an essential element of the gain medium of interband cascade lasers (ICLs) emitting in mid-infrared spectral range. The ICLs have been proven to have a tremendous application potential in optical gas sensing of medically and environmentally relevant gasses and overwhelming in some performances the systems based on quantum cascade lasers. In spite of that, some of the properties of the active region in this experimentally demanding spectral range are still not well known, and this also concerns carrier dynamics, on which only a very initial work has been reported. In this work, we use experimentally challenging pump-probe technique utilizing pJ pulses to study at carrier dynamics at room temperature in the W-type QWs to get a set of characteristic laser-performance-related time constants.

The transient reflectivity (TR) experiment based on a pump-probe scheme has been used to test carrier relaxation dynamics in such a W-design OW structure. The OW is excited by a train of 140 fs-long pulses with the photon wavelength of 830 nm, and ~13.12 ns pulse-topulse distance that produces certain carrier population in the higher energy states in the well. Subsequent carrier relaxation is tested by a train of ~250 fs-long probe pulses that are tuned to the ground state (GS) emission of the OW occurring at 2.5 µm. The TR signal reveals two characteristic processes: (i) population of the GS hindered in the rise of the TR signal, and (ii) its subsequent depopulation represented by the decay of the transient reflectivity amplitude. The measured TR rise time is 2.3±0.2 ps, and it is supposed to be related to the longitudinal phonon-assisted relaxation channel since the initial population of photo-injected carriers is insufficient for the Auger-type relaxation. The GS depopulation occurs via two processes of a significantly different time scale. The long-lasting one in the nanosecond range is attributed to the radiative recombination of spatially separated electrons and holes - expected for the Wtype QW structure. However, the origin of a short decay component of 240±0.2 ps time constant is not clear and has not been observed for such type of OWs. It can be attributed to the carrier escape process from the QW GS to some localized states at the InAs/GaInSb interfaces caused by the intermixing effect, which has been predicted previously.

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## Open-Path High Precision Sensor for Urban NO, NO<sub>2</sub> and NH<sub>3</sub> Detections

Chenguang Yang 1, Erchao Niu2, Ruifeng Kan 1 and Mai Hu 1

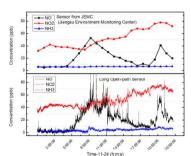
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Despite their low concentrations, nitric oxide (NO) and nitrogen dioxide (NO<sub>2</sub>) represent an important factor regarding air quality by local ozone production and secondary organic aerosol formation [1]. Atmospheric ammonia (NH<sub>3</sub>), the third most abundant nitrogen species and dominant atmospheric base, is an important precursor to ammoniated fine particulate matter [2]. High sensitivity gas phase measurements are necessary for local air pollution prediction and analysis of haze formation [3].

For the present study, we adopted medium infrared absorption spectrum with wavelength modulation and open-path technologies for atmospheric NO, NO<sub>2</sub> and NH<sub>3</sub> detection.



**Fig. 1.** Picture of Open-path NO, NO<sub>2</sub> and NH<sub>3</sub> Sensor



**Fig. 2.** Ambient monitoring of NO, NO<sub>2</sub> and NH<sub>3</sub>

Three absorption lines located at 1900 cm<sup>-1</sup>, 1600 cm<sup>-1</sup> and 1103.4 cm<sup>-1</sup> were selected for NO, NO<sub>2</sub> and NH<sub>3</sub> detection, respectively.

Three QCLs were multiplexed using beamsplitters, and expanded six times to reduce divergence angle. The reflected beam from the reflector was focused on a room-temperature MCT photodiode by an off-axis parabolic mirror and precise adjustment was made to optimize the detector signal. The optical path of the sensor can be adjusted from 100 m to 2km.

The MDL results are less than 0.1 ppb\*2km for three gases within 10 second minute integral time.

As shown in Figure 2, the open-path sensor has the similar daily variation with the sensor from Jiangsu Environment Monitoring Center, which uses chemiluminescence technology, and it has much higher precision and timing resolution.

#### Acknowledgement

The work is supported by National Key Scientific Instrument and Equipment Development Project of China (2014YQ060537)

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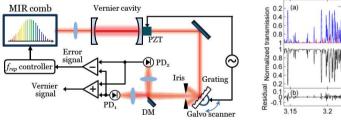
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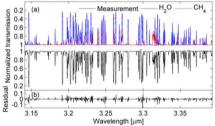
#### **Po12**

## High-power Broadband Mid-Infrared Frequency Comb Sources for Vernier Spectroscopy

G. Sobon 1,2, A. Khodabakhsh 1, L. Rutkowski 1, Chuang Lu 1, and A. Foltynowicz 1

Mid-infrared (MIR) optical frequency combs enable simultaneous detection of many molecular species at low concentrations. Here, we demonstrate a fast, robust and sensitive spectrometer based on continuous Vernier filtering of a MIR comb by an enhancement cavity that allows detection of 2 ppb of CH<sub>4</sub> in 25 ms [1]. The comb source is a doubly-resonant optical parametric oscillator (OPO) based on an orientation-patterned GaAs crystal synchronously pumped by a Tm: fiber femtosecond laser [2]. The signal comb (3.1-3.4 µm, 30 mW at 125 MHz repetition rate, free) is coupled to an enhancement cavity with a finesse of ~350 and free spectral range of 250 MHz [Fig. 1(a)]. By slightly detuning the cavity length from the perfect match length ( $L_{PM} = c/2f_{rep}$ ) the cavity resonances act as a filter and transmit groups of comb modes called Vernier orders. A diffraction grating mounted on a galvo scanner separates these orders after the cavity and one order is imaged on two photodiodes (PD). The Vernier order is tuned across the comb spectrum by scanning the cavity length and fixed in space by synchronous rotation of the grating, allowing the acquisition of the entire signal range in 25 ms. An error signal derived from the difference of the PD outputs is used to actively lock the scan of the Vernier order and the grating rotation. A normalized transmission spectrum of laboratory air containing 2.08 ppm of CH<sub>4</sub> and 0.641% of water is shown in Fig. 1(b). The red and blue curves show a fit of the model spectra calculated using the line parameters from the HITRAN database.





**Fig. 1**. (a) Experimental setup of the Vernier spectrometer: PD<sub>1,2</sub> – HgCdTe detectors, DM – D-shaped mirror. (b) Normalized Vernier spectrum of laboratory air (black, 6.8 GHz resolution) with 2.08 ppm of CH<sub>4</sub> and 0.641% of water along with a fit of the model spectra of CH<sub>4</sub> (red) and H<sub>2</sub>O (blue), and residual (below).

The sensitivity of the spectrometer,  $1\times10^{-9}$  cm<sup>-1</sup> Hz<sup>-12</sup> per spectral element, is detector noise limited, so it can be improved by increasing the power of the MIR source. For this purpose, we designed and developed a new comb source based on difference frequency generation (DFG) in a periodically poled lithium niobate crystal, pumped by a femtosecond Yb:fiber laser. This DFG source is tunable in the 2.7–4.2  $\mu$ m range and delivers 230 mW of power in ~200 nm bandwidth around 3.3  $\mu$ m [3]. The use of the DFG source instead of the OPO will reduce the complexity of the Vernier spectrometer and the high output power will translate into increased sensitivity.

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## Resonant Tunneling Diode Photodetectors for Light Detection at the Telecommunication Wavelength of $\lambda=1.3~\mu m$

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We have investigated the electro-optical transport properties of resonant tunneling diode (RTD) photodetectors for light detection at the telecommunication wavelength of  $\lambda=1.3~\mu m$ , and identified the physical factors that determine and shape the RTD photoresponse.

Due to their aptitude to amplify optically generated charge carriers, resonant tunneling diodes (RTDs) are increasingly deployed and investigated as photodetectors.[1], [2] Extensive work has been done to demonstrate single photon detection, [3] and even photon counting, [4] yet some dynamic effects that shape the RTD photocurrent-voltage relation itself have remained uncharacterized. We present a detailed study on the photocurrent-voltage relation and the accumulation dynamics of photo generated minority charge carriers (holes for n-type RTDs) in RTDs. The RTDs were grown by molecular beam epitaxy on an n-type Si-doped GaAs substrate. The RTD double barrier resonant tunneling structure consists of two 3 nm Al $_{0.6}$ Ga $_{0.4}$ As barriers embedding a 4 nm GaAs quantum well. The 158 nm wide GaInNAs absorption layer was grown lattice-matched to GaAs with a bandgap energy of  $E_g \cong 0.95$  meV. RTD mesa structures with a diameter of 5  $\mu$ m were fabricated by standard lithographic processes and dry chemical etching techniques. The bottom contact on the backside of the substrate was formed by an alloyed Ni/AuGe/Ni/Au contact. A Ti/Pt/Au ring-shaped contact was deposited on top of the RTD mesa, which guarantees optical access and electrical contact.

We find that at negative and small positive voltages, the photocurrent is negligible small due to a low quantum efficiency  $\eta(V)$ . At higher voltage, the RTD photocurrent is limited by a decreasing lifetime  $\tau(V)$  of photogenerated holes confined at the resonant tunneling structure (RTS). [5] At low light levels, the RTD shows a constant sensitivity and the photocurrent increases linear with incident illumination power. For higher powers, a feedback mechanism due increased hole accumulation reduces the lifetime and thus decreases the sensitivity. [6]

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## APOSEMA - Advanced Photonic Sensor Materials for Breath Analysis

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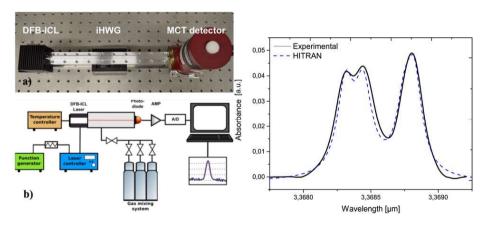
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Exhaled breath analysis is used to non-invasively obtain information on the clinical state of an individual by monitoring exhaled (bio)markers, which are either endogenously produced or administered and subsequently metabolized within the human body. To achieve high selectivity and sensitivity, typically technically sophisticated and expensive analysers are needed limiting their implantation at the physician office. In order to capitalize on the potential of breath analysis for identifying particular medical conditions, innovative analytical methods need to be developed, i.e., technology must be reduced in size and complexity to meet day-to-day clinical requirements. The aim of the project APOSEMA – a collaboration of Austrian and German academic and industrial partners – is to detect different types of trace gases rapidly and reliably via innovative measurement methods. A new type of highsensitivity sensor system combining fluorescent and infrared detection for exhaled breath and gas analysis in a single device is developed providing fast and reliable trace gas analysis probing possibly small gas sample volume. The developed device is based on nano-hybrid materials for oxygen detection and analyte enrichment, interband cascade lasers (ICLs) as mid-infrared light source, and substrate-integrated hollow waveguides (iHWGs) serving as IR photon conduit and miniaturized gas cells. As an example [1], we demonstrate that the developed sensing platform prototype enables the determination of ppm-level concentrations of methane in exceptionally low volume samples (i.e., few hundred microliters).



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# Sensing Systems III

## Recent Progress in Trace Gas Instrument Development at Aerodyne Research, Inc.

#### J.B. McManus, D.D. Nelson, C. Dyroff, and M.S. Zahniser

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We present several avenues of ongoing development of laser spectroscopic trace gas instrumentation at Aerodyne Research, Inc (ARI). We currently offer two commercial instrument platforms for trace gas measurements, a single laser and a dual laser instrument. These instrument platforms, with appropriate choices of laser(s), detector and multi-pass cell offer a wide variety of possible trace gas measurements, at the highest levels of precision. The lasers, operating in the mid-infrared (~3 to 11 μm) may be either quantum cascade lasers or interband cascade lasers. ARI has recently produced instruments that measure combinations of trace gases, including: {N<sub>2</sub>O, CO<sub>2</sub>, CO, H<sub>2</sub>O}, {N<sub>2</sub>O, CH<sub>4</sub>, H<sub>2</sub>O}, {NH<sub>3</sub>}, {C<sub>2</sub>H<sub>6</sub>}, {COS, CO<sub>2</sub>, H<sub>2</sub>O}, {HCHO, HCOOH}, {NO, HONO}, {HNO<sub>3</sub>}, {SO<sub>2</sub>}, {CO<sub>2</sub>, CO<sub>2</sub>, CO<sub>2</sub>, CO<sub>3</sub>, {CO<sub>4</sub>, H<sub>2</sub>O}, {CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>5</sub>, CO<sub>6</sub>, {CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>6</sub>, {CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>6</sub>, {CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>6</sub>, {CO<sub>4</sub>, CO<sub>4</sub>, CO<sub>6</sub>, {CO<sub>6</sub>, {CO<sub>6</sub>, CO<sub>6</sub>, {CO<sub>6</sub>, CO<sub>6</sub>, {CO<sub>6</sub>, CO<sub>6</sub>, {CO<sub>6</sub>, CO<sub>6</sub>, {CO<sub>6</sub>, {CO<sub>6</sub>, CO<sub>6</sub>, {CO<sub>6</sub>, {CO<sub>6</sub>, {CO<sub>6</sub>, CO<sub>6</sub>, {CO<sub>6</sub>, {CO<sub>6</sub>,

	Conc.	1	OPL	Absorption	1s N	loise:
Gas	ppb	cm	m	Depth	ppt	absorption
$N_2^{O}$	320	2200	76	0.15	3.4	1.7*10 <sup>-6</sup>
COS	0.5	2040	210	6.2*10 <sup>-4</sup>	4.5	1.4*10
$^{13}CO_2$	3800	2310	7.3	0.12	800	2.4*10 <sup>-6</sup>
$^{13}CH_{4}$	18	1294	210	2*10 <sup>-3</sup>	25	3*10 <sup>-6</sup>

Recent results include record low instrument noise. For example, measurements of carbonyl sulfide (COS) in the air (0.5 ppb) has 1s noise of  $\sim$ 4 ppt and reached 0.22 ppt with 1000 s averaging. That corresponds to a minimum absorption noise of  $\sim$ 1.5x10<sup>-7</sup>. Improvement in instrument performance comes in part from improved understanding of noise sources and the propagation of noise through the measurement system.

In addition to our standard instruments, we are developing several other platforms. One new platform is a two-laser instrument that is the same size as our present single laser instrument. Initial tests of that instrument show performance comparable to our standard instruments. We are also working on a miniaturized instrument for application to small mobile platforms, such as unmanned aerial vehicles. The miniaturized system utilizes a small lens for light collection and beam forming. This has allowed us to observe fascinating laser feedback phenomena.

The application of QCL and ICL based trace gas instruments to environmental measurements helps to provide much needed answers to pressing environmental questions. For example, measurement of methane emissions to the atmosphere from energy industry operations helps in evaluation of national greenhouse gas budgets and helps to guide methods to reduce those emissions. Measurement of fluxes of gases helps to quantify the exchange of gases between the environment and the atmosphere. Measurement of very rare gases, such as nitric acid, hydrogen peroxide, nitrous acid, in combination with the measurement of many other gases, helps to clarify basic atmospheric chemistry processes.

#### Tul4

#### INVITED

## MIR TDLAS Technology for Industrial Emission and Environmental Monitoring

#### Ruifeng Kan, Xueli Fan, Chenguang Yang, and Bing Chen

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Thanks to the development of new lasers in MIR such as interband cascade laser, quantum cascade laser, TDLAS technology has been applied wider and wider in environmental, industrial, biological and other areas. For the last decade, our group has been focusing on TDLAS technology, and developing many kinds of instrument with NIR/MIR TDLAS. Several research topics about MIR TDLAS technology for industrial emission and environmental monitoring in our group will be presented in this talk.

Global warming has been one of the most attention issues on the earth. For measuring  $CO_2$  vertical distribution of troposphere, we built a small compact TDLAS instrument with 0.1 ppm detection limit. Mounted on the balloon and glider respectively, the instrument measured  $CO_2$  vertical concentration distribution in Mongolian Steppe and Taklamakan Desert [1]. Besides measurement of  $CO_2/CO/CH_4$  with NIR TDLAS, we measured  $CH_4$  and  $N_2O$  with one cw-QCL. By calibration-free WMS method, the detection limits of the sensor are 4 ppb for  $CH_4$  and 2 ppb for  $N_2O$  [2]. We also are studying on Carbon Dioxide Isotope Monitor with MIR TDLAS technology.

Recent years, haze has become the most serious environmental issue in China. Despite their low concentrations, NO, NO<sub>2</sub> and NH<sub>3</sub> represent important factors regarding air quality by local ozone production and secondary organic aerosol formation. For the present study, we adopted MIR-WMS technologies for atmospheric NO, NO<sub>2</sub> and NH<sub>3</sub> detection. Two kinds of detections are developed, based on open-path multipass-cell (60m) and long open-path (2km), detection limits of which are below than 0.5 ppb and 0.1 ppb respectively for all three trace pollutions [3].

In the near future, odors will be included in air quality monitoring system. Responding to odor monitoring requirement of the environment officials, we have started researching on MIR sensor for all eight odors in Chinese national standard, including ammonia, hydrogen sulfide, methyl mercaptan, dimethyl sulfide, dimethyl disulfide, carbon disulfide, trimethylamine and styrene.

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## High Performance Spectroscopy of Hydrocarbon Gas Mixtures in the 6 – 11 µm Range

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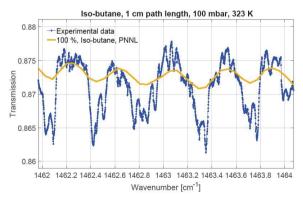
Gas chromatographs are widely employed to measure the composition of hydrocarbon process streams as they offer the capability of measuring a multitude of components. Concurrently, chromatographs exhibit disadvantages as limited response time and sophisticated calibration requirements. Meanwhile, quantum cascade lasers are on a stage of development revealing high resolution, highly sensitive and real-time measurements for gas sensing in the mid-infrared. Hence, the spectroscopic determination of hydrocarbons and their mixtures provides access to new applications for improved process control in oil-refining industries.

Available spectra from standard databases as HITRAN and PNNL do not provide the required spectral resolution for the entire set of hydrocarbons of interest (C<sub>1</sub>-C<sub>5</sub> alkanes including isomers) under application specific conditions for pressure and temperature.

Therefore, we present the development of an external cavity quantum cascade laser based setup that is designated for the operation as laboratory reference system in order to thoroughly determine the reference spectra of individual hydrocarbons and their mixtures. The setup allows a broad spectral coverage from 6-11  $\mu$ m and the exact control of gas pressure and temperature during the measurements. Measurements with a spectral accuracy of at least  $3 \cdot 10^{-3}$  cm<sup>-1</sup> and a "per point" minimum detectable absorption of  $1.3 \cdot 10^{-4}$  Hz<sup>-1/2</sup> could be acquired. Beside the optical setup a sophisticated gas sampling unit was developed providing the capability of creating accurate real gas mixtures of arbitrary composition.

The measurements are suitable to study broadening effects on the spectra of gas mixtures that arise from variations in pressure, temperature, concentration and the individual gas components. An exact determination of these influences is mandatory to guarantee reliable sensing of all gas species involved. We will present latest measurement results highlighting the increased spectral resolution

The project has received funding from the European Union's Horizon 2020 research and innovation program under grant agreement No 636930.



**Figure 1:** Measured transmission spectrum of iso-butane and respective scaled PNNL spectrum for a temperature and pressure of 323 K and 100 mbar, respectively. The structure of the measured curve demonstrates the increase in resolution of the measurements compared to the database.

#### TuO8

## Breath Alcohol – High Precision Measurement of VOCs Using a DFB-QCL

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Volatile organic compounds (VOC) are ubiquitous from both anthropogenic and natural sources. Therefore, their selective and precise detection is of great interest in many industrial, environmental, medical, and forensic applications. Measurement of breath ethanol (EtOH) is particularly important since it is the most widely employed forensic science procedure.

As nearly all organic molecules, EtOH has intrinsically broad and congested absorption features, which make its quantification by mid-IR laser spectroscopy challenging. Several broadly tunable MIR laser technologies can provide light sources for suitable spectroscopic instrumentation. Most popular is the External Cavity (EC) QCL which, however, is limited by relatively slow tuning, mode hops, sensitivity to mechanical vibrations, and price. Highly promising are recently published Extended Tuning (ET) [1] and Very Large Tuning (XT) [2] QCLs, which provide relatively wide, fully electrical tuning, fast scanning, and high spectral resolution typical for DFB lasers. However, they are currently only available at selected frequencies.

Here, we demonstrate how a conventional DFB-QCL can efficiently be used for selective and sensitive measurement of EtOH in a breath-like gas matrix (including 5.0 % of CO<sub>2</sub> and 5.4 % of H<sub>2</sub>O). This is possible due to the strong absorption and sharp spectral features at 9.3 μm in spectra recorded under reduced gas pressure, which can easily be resolved by the DFB-QCL. To enlarge the tuning range and minimize heat dissipation of the laser we use the intermittent *cw* operation [3]. An open source FPGA based 14 bit A/D converter with up to 125 MHz time resolution provides fast data acquisition and controls the laser driver [4].

The developed instrument provides low ppb precision at concentrations up to 250 ppm EtOH as required for metrological studies investigating reference gas generation methods for breath analysis in traffic related breath tests. The general approach however can be applied to a wide range of other VOC measurements.

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## TDLAS Determination of Carbon Dioxide Isotope Ratio for Diagnosis of Helicobacter Pylori

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The measurement of <sup>13</sup>CO<sub>2</sub> to <sup>12</sup>CO<sub>2</sub> isotope ratio in human breath is involved in one of the standard methods used for the diagnosis of infection from Helicobacter Pylori, a bacteria living in the gut and linked with many gastric diseases such as ulcer and cancer. The measurement is done before and after a test meal enriched with urea containing the less abundant isotope <sup>13</sup>C. As urea is promptly processed by Helicobacter Pylori in the stomach, positive subjects will show an increased level of <sup>13</sup>CO<sub>2</sub> in their exhaled breath.

The required threshold for discrimination between positive and negative patients is about 3 per mille in the isotope ratio.

While the measurement is currently done with other techniques such as mass spectrometry and optical spectroscopy in the mid infrared, Tunable Diode Laser Absorption Spectroscopy (TDLAS) in the  $2\mu m$  band is a possible candidate for the development of a simple, stable, compact and inexpensive testing instrument.

In this work the choice of spectral lines and proper signal evaluation algorithms are presented together with the instrument design, all critical aspects due to the high dynamic range required for the measurement.

Moreover, long term stability and drifts of the system may be important if the instrument will be intended to operate not only on collected samples but also on a live breath sampler.

Preliminary tests on breath sample cells operating at low pressures, with different lengths and different configurations (either linear or multipass) are shown in order to prove the feasibility of this method and fully characterize the available performances obtainable with different arrangements.

### 17 May (Wednesday)

#### Sessions

Sensing systems IV Laser sources III Laser sources IV



**Wrocław Town Hall** 

### In-situ H<sub>2</sub>S and SO<sub>2</sub> Tail Gas Analysis with Near- and Mid-infrared TDLS

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NEO Monitors AS, Prost Stabels vei 22, 2019 Skedsmokorset, Norway
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 Norsk Elektro Optikk AS, Prost Stabels vei 22, 2019 Skedsmokorset, Norway

Elemental sulfur is an important component for many industries, especially for the manufacture of fertilizers. The *Claus process* is one of the most commonly used processes to recover elemental sulfur from gaseous hydrogen sulfide (H<sub>2</sub>S) which is present in numerous gaseous waste streams from, for example, natural gas plants and oil refineries. Tail gas analysis (TGA) is an important part of the recovery process to increase efficiency and lower emissions of H<sub>2</sub>S as well as sulfur dioxide (SO<sub>2</sub>); the latter is generated during the conversion process. Traditionally, TGA was performed by gas chromatographs (GCs). In recent times, the majority of TGA is done by heated UV analyzers. While the measurement setups are fairly straight forward, they have high demands on maintenance and the generated sulfur is frequently clogging the extraction pipes.

Tunable diode laser spectroscopy (TDLS) is nowadays considered to be a mature technology for in-situ measurements allowing to perform measurements – even in harsh environments – with a higher accuracy and faster response time than extractive methods while requiring significantly lower maintenance. This success, especially taking aforementioned issues with extractive systems into account, has led to the sulfur recovery industry's interest in evaluating a similar technology for in-situ measurements of H<sub>2</sub>S and SO<sub>2</sub> for TGA.

In-situ measurements of  $H_2S$  with TDLS have been demonstrated in many different applications using lasers in the NIR spectral region. Until recently, laser-based measurements of  $SO_2$  were considered to be impossible since  $SO_2$  does not have any absorption bands with sufficient strength in the NIR. In the MIR, however, several strong absorption bands are available. The advent of interband cascade lasers (ICLs) has opened up the opportunity to extend the measurement capabilities of TDLS from the near- to the mid-infrared region and, thus, enabled also the measurement of  $SO_2$  [1].

Following a laboratory characterization, to our best knowledge for the first time, in-situ TDLS measurements for TGA in a German sulfur recovery plant with a  $H_2S$  sensor operating in the NIR and a  $SO_2$  sensor operating in the MIR were performed. During the measurement campaign, a co-located extractive UV analyzer was used as reference system.

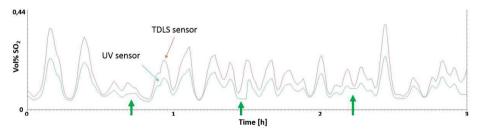


Figure 1 Comparison of SO<sub>2</sub> concentration measurements with UV and TDLS sensors.

Excellent agreement between both measurement systems was achieved (Figure 1), thus demonstrating that it is feasible to regulate a Claus process with in-situ instrumentation.

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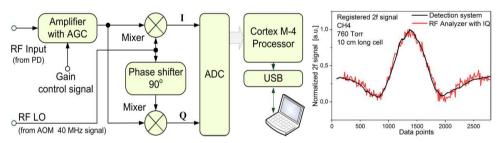
#### WeO2

## IQ Demodulation-based Gas Sensing Detection System for the Photothermal and Chirped Laser Dispersion Spectroscopy

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In this paper we present latest comparative results on using custom-designed, compact and low-cost detection system for a gas sensing, especially dedicated for the Photothermal [1] and Chirped Laser Dispersion Spectroscopy (CLaDS) [2] methods, where FM signal demodulation is needed to get an information about gas concentration level. The IQ demodulator is most sensitive circuit to FM signal demodulation. Some expensive RF analyzers offer a special IQ extension modules and they have been used to gas sensing as a demodulator in CLaDS method [3]. The presented detection system provides an alternative to RF analyzer-based gas sensing setup and it offers about 20-times less amplitude fluctuation of measured 2f signal at the same averaging level against to detection setup with RF analyzer. Additionally, it allows to build a fully integrated, mobile and compact laser gas spectroscopy system, especially fiber-based setups. The gas sensing detection setup used to detect CH<sub>4</sub> particles enclosed in a gas cell by photo-thermal spectroscopy method and comparative results of weak 2f demodulated signals are presented in Fig. 1.



**Fig. 1** Detection system setup (left) and comparison of the registered 2f photo-thermal weak signals by the presented system (black line) and RF analyzer (red line). AGC – automatic gain control, ADC – analog to digital converter, AOM – acousto-optic modulator, PD– photodiode.

The carefully designed RF amplifier with AGC and low distortion Mixer circuit provides less signal noise after demodulation due to stable IQ signals amplitude. Both IQ signals are sampled simultaneously by ADC where inputs duplication technique for the same converted signal is used and it provides extra 6 dB higher signal to noise ratio (SNR).

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#### Acknowledgments

We acknowledge support from the National Science Centre within the project DEC-2014/14/M/ST7/00866.

## Compact and Lightweight Multipass Cell Designs with Optimized Beam Propagation

#### M. Graf, B. Tuzson, and L. Emmenegger

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Compact and lightweight instruments for the accurate measurement of trace-gas concentration are of great importance. The advent of quantum cascade lasers was an important trigger for the development of mobile and field-deployable mid-IR spectrometers [1]. Another key element is the multipass cell (MPC), which is most frequently based on the designs of White [2] and Herriott [3]. Recently, monolithic circular designs have received significant attention, because of their potential with respect to optical path, size, weight, and ruggedness, especially in the context of mobile and lightweight devices.

This work presents a theoretical and computational method to investigate the laser beam propagation within monolithic toroidal multipass cells [4, 5]. Numerical electromagnetic field calculations are presented, which reveal the optical characteristics and fundamental limitations of this MPC-design and suggest optimal configurations.

Furthermore, we propose a novel concept for circular MPCs that overcome most of the current limitations. By adjusting the local curvature of the inner surface, the inherent rotational symmetry of a toroidal ring is discretized. This segmentation can be used to obtain a confocal cavity configuration which ensures optical stability of the laser beam [6]. As a result, efficient beam folding is realized, optical interferences are reduced, and the transmission is maximized. Moreover, a collimated laser beam can be directly coupled into the cell without the necessity of being shaped with additional optical elements. Thus, very minimalistic optical setups are possible, involving only a laser, a detector and the MPC. Preliminary tests comprising a MPC-prototype with 6.6 m optical path length indicate a 10<sup>-3</sup> noise level. Several strategies to further enhance the performance have been identified, which will be discussed and experimentally verified. Since this versatile MPC-concept can support more than 10 m optical path length in a small detection volume (< 140 ml) and a total weight of 200 g, it is a promising solution for size- and mass-critical applications in both open and closed configuration.

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#### WeO4

## **Temperature Measurements from 2μm Carbon Dioxide Absorption Spectrum**

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In the latest years many instruments for non-invasive carbon dioxide sensing have been developed and found their way to the industrial market for in line measurement inside close containers such as sealed packages and bottles.

One of the main requests from the end users is measurement of pressure and concentration in carbon dioxide enriched headspaces. This can be done by analysis of the absorption spectrum in the 2µm region through Tunable Diode Laser Absorption Spectroscopy (TDLAS).

An additional useful parameter often asked together with the carbon dioxide measurement is the temperature of the gas. This is interesting both as a process parameter itself as well as to establish the relationship between carbon dioxide in the gas phase and dissolved into liquid and solid media inside the sample.

From a spectroscopic point of view, absolute temperature can be evaluated by fitting against the Boltzmann model the measured absorption strength ratios between lines originating from different energy levels. Such lines, spanning initial energy levels from 234 to 772 cm<sup>-1</sup>, can be found within a single spectral scan of a VCSEL diode (which is capable of a few nanometers detuning through modulation of injection current), and are actually the same used for typical carbon dioxide concentration and pressure measurement in commercial sensors.

In this work a TDLAS test bench was set up with a 15cm long gas absorption cell in order to test the sensitivity and accuracy of such an absolute temperature measurement during slow transients between 30 and 60°C. The measurements were repeated at different pressures and concentrations to simulate some typical sample environments, spanning from 0.5 to 2 bar abs. and from 25 to 100% carbon dioxide concentration.

From a performance point of view, resolution and repeatability better than 1°C have been shown, with a total accuracy on the absolute temperature measurement better than 5°C without any instrument calibration other than the knowledge of absorption line parameters (from HITRAN spectral database). The accuracy can be even improved with just a simple (1 point) calibration to take into account the global effect of all the deviations from the theoretical model.

#### **Upconversion Detection for Gas Sensing Applications**

#### Y.-P. Tseng, L. Meng, P.J. Rodrigo, C. Pedersen, and P. Tidemand-Lichtenberg

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Laser-based gas sensing devices have found a wide range of applications, such as hazardous gas detection [1], exhaled breath analysis [2] and food inspection [3]. Spectral absorption features in the infrared (IR) region from 1.5 to 15  $\mu$ m can be used for sensitive detection of molecular structures in a unique way that allows for accurate identification and quantification of chemical compounds. However, the existing IR detectors – depending on the spectral range of interest – are mainly based on indium gallium arsenide (InGaAs) or mercury cadmium telluride (MCT) that have typically lower response time and detectivities several orders of magnitude lower than that of silicon detectors. It has been previously demonstrated how the IR radiation can be detected via nonlinear frequency upconversion which allows for the use of silicon detectors to acquire upconversion signal in visible or near infrared ranges [4]. In this paper, the upconversion approach is presented and exemplified by two specific systems for gas detection in the 1.5  $\mu$ m and 10  $\mu$ m ranges, respectively.

In the region below 5  $\mu$ m, periodically poled LiNbO<sub>3</sub> (PPLN) is one of the preferred nonlinear materials due to its high nonlinear efficiency, low loss and tailorable phase-matching properties. Figure 1(a) shows the PPLN-based upconversion detector used in an integrated-path differential absorption lidar system [5] for atmospheric CO<sub>2</sub> monitoring. The backscattered on-line and off-line signal at 1575 nm is upconverted to 635 nm by mixing with a 1064 nm pump laser in a 2.5 cm long PPLN crystal. The upconverted signal is measured by a photomultiplier tube instead of measuring the backscattered 1575 nm signal directly. An intrinsic upconversion quantum efficiency of 60% is achieved experimentally. We note that PPLN has attractive properties such as a large nonlinear coefficient of  $d_{\rm eff} \sim 16$  pm/V, but it cannot be used beyond 5  $\mu$ m.

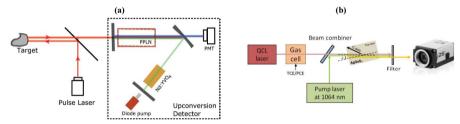


Fig. 1. Upconversion systems for (a) CO<sub>2</sub> monitoring and (b) for TCE detection.

Moving to longer wavelengths, there is a limited number of commercially available nonlinear crystals. In this work a silver gallium sulfide (AgGaS<sub>2</sub>) crystal is used. AgGaS<sub>2</sub> is a good candidate for upconversion of long-wave IR radiation because of its broad transparency range going up to 12  $\mu$ m, large nonlinear coefficient of  $d_{\rm eff}$  ~16 pm/V and being commercially available. Figure 1(b) shows the setup for upconversion spectroscopy in the 10  $\mu$ m range. The narrowband tunable output (9  $\mu$ m to 12  $\mu$ m) from a quantum cascade laser (QCL) is passed through a Herriott gas-cell containing the gas (e.g. TCE or PCE) to be measured. The IR radiation transmitted through the gas-cell is mixed with a 1064 nm laser followed by detection using a standard silicon detector. The phase-matching condition at each wavelength can be obtained by angle-tuning of the AgGaS<sub>2</sub> nonlinear material [6].

In both systems, superior performance is achieved when compared to direct detectors in the IR.

#### Funding Mid-TECH H2020-MSCA-ITN-2014 Grant agreement no.: 642661

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#### WeO6

## Stable Isotope Analysis of <sup>13</sup>CH<sub>4</sub> and CH<sub>3</sub>D In Mixed Biogenic and Fossil Methane Samples

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The renewable energy directive 2009/28/EC [1] promotes the use of renewable energy sources. There is a need for reliable biofraction determination of an unknown methane sample to prevent misuse of the market position of renewables compared to natural gas. In addition, isotopic composition of the produced biomethane carries information of the production process viability and provides an early warning of any process failure [2].

We have developed a tunable diode laser spectrometer (TDLS) for simultaneous measurement of  $^{13}\text{CH}_4$  and  $\text{CH}_3\text{D}$  in methane samples. A mid-infrared Interband Cascade Laser emitting at 3.267  $\mu\text{m}$  is used to measure the absorption of the three most abundant isotopologues in a single current sweep. A multipass-cell with a 1 m pathlength and < 20 mL sample volume resulted in sufficient absorption depth for all isotopologues. A Voigt profile was fitted in real time to the measured absorption profiles. Two methane samples with different isotopic composition were used as working standards to correct for instrumental drift. Biogenic methane samples originating from three different sites in Finland were analysed. In addition, three mixtures of fossil and biogenic methane with different biofractions were prepared and analysed with the developed instrument to assess the usability of stable isotope analysis for biofraction determination with and without pre-knowledge of the methane sources.

The final referenced measurement precision was less than 1‰ for both isotopologues. Measurement uncertainties and their sources were identified. The measured biofraction ratios of the mixed samples correlated well with the actual mixing ratios of the volumetrically prepared mixtures. There were notable differences in isotopic ratios of biogenic samples from different sources. This limits the accuracy of the biofraction determination with stable isotopes in the case where origin of the mixed gases is unknown. The performance of the developed methane isotope analyser for on-line and real-time measurement was tested; the instrument demonstrated its applicability for precise long-term monitoring of biogas production process.

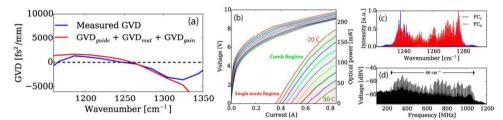
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## Waveguide Engineering for Low Dispersion Mid-Infrared Quantum Cascade Lasers Frequency Combs

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Quantum Cascade Lasers (QCLs) have become one of the most used light sources in the Mid-IR. It has been demonstrated that QCLs can operate as frequency combs (FCs) and that the performance of QCL FCs can be significantly improved by compensating for their waveguide dispersion using dielectric coating based Gires-Tournois interferometers [1]. However, the latter are incompatible with high optical output power since due to the optical absorption of the materials they overheat and burn. In this work we investigate how the properties of mid-IR QCL FCs can be improved by tailoring their waveguide dispersion. A QCL with an active region based on a single stack double-phonon QCL design emitting at 7.8 µm was grown. A plasmon enhanced waveguide was used and its dispersion tailored in order to reduce the group velocity dispersion (GVD). A 10.5 µm wide device was cleaved to a 3 mm long bar. The GVD of the device was measured using the so called Fourier-Transform technique [2] and is displayed in Fig. 1 (a) (blue line) showing a good agreement with the computed value. At 1250 cm<sup>-1</sup> the measured GVD is -42 fs<sup>2</sup>/mm.



**Fig. 1:** (a) Measured and calculated GVD of the device (b) Light-current-voltage curve of the device. (c) Optical spectra of the two combs at -15 °C for a current of 800 mA (FC<sub>1</sub>) and 700 mA (FC<sub>2</sub>). (d) Corresponding multi-heterodyne spectrum.

The output power of the device was then measured as a function of current and voltage for temperature ranging from -20 C to 50 °C and reached 180 mW as shown in Fig. 1 (b). For all operating points beatnotes narrower than 750 Hz were measured using a bandwidth of 200 Hz (Fig. 1 (d)). The optical spectra at -15 °C is displayed in Fig. 1 (c), demonstrating that the optical frequency span of the FC reaches 60 cm<sup>-1</sup>. In order to demonstrate the capabilities of the fabricated devices for dual-comb spectroscopy, the multi-heterodyne beating spectrum of two of the devices was measured and is displayed in Fig. 1 (d). 93 modes are measured demonstrating that the dual comb spans 46 cm<sup>-1</sup>.

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#### WeO8

#### **Tuning Quantum Cascade Laser Wavelength by the Injector Doping**

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The well-known feature of quantum cascade lasers (QCLs) is the sensitivity of emitted radiation wavelength to the temperature and bias conditions. [1] Much less known possibility of wavelength tuning concerns its variation with the doping level of active wells. While, for the obvious reasons, this method cannot be used in precise tuning of QCL-devices mounted onboard the sensing heads it seems attractive for the preselection of spectral range while designing devices dedicated for specific applications. In this contribution we demonstrate this possibility both experimentally and theoretically. Our main result is presented in Fig. 1, where the lasing characteristics of several QCL devices (GaAs/Al<sub>0.45</sub>Ga<sub>0.55</sub>As 3-well active region design [2]) doped (two well-barriers pairs in the central part of the injector) to different densities are shown [1]. The wavelength-doping correlation is clear.

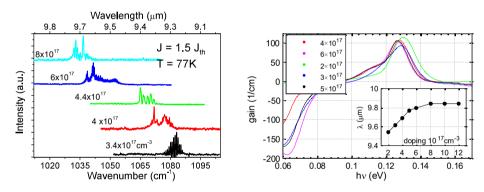


Fig. 1 (left) Emission spectra of GaAs QCLs doped to different densities. The lasers are otherwise identical. (right) Calculated gain spectra and gain peak wavelength λ vs. doping (inset)

As we deal with stratified unipolar n-type device our theoretical analysis uses single-band effective mass 1D Hamiltonian parametrized for the in-plane kinetic energy, solved self-consistently with the Poisson equation. Simulations of the devices are made with non-equilibrium Green's function (NEGF) method in the real space [3]. Calculations comprise scattering with phonons, ionized impurities, interfaces roughness and alloy disorder. At the output of the calculations gain spectra are calculated for variously doped devices. In general, good qualitative agreement between the values of the gain peak frequency hv and the experimental wavelength  $\lambda$  is found. Inspection of the data allow to interpret the observed effect in terms the doping-originated Stark-shift. Simulations reveal also that the observed wavelength vs. doping dependence can only be observed in the limited doping range.

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## GaSb SLDs and gain-chips for sensing applications in the 2-2.5 micron wavelength range

I. Šimonytė<sup>1</sup>, R. Wang<sup>2</sup>, K. Vizbaras<sup>1</sup>, G. Roelkens<sup>2</sup>, and A. Vizbaras<sup>1</sup>

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In this work we will present latest results on development of high-performance GaSb type-I based superluminescent diodes (SLDs) and broadband gain-chips for spectroscopy and sensing applications in the 2-2.5 micron wavelength range. This wavelength range is of particular interest for many applications due existence of numerous gas and biological substance absorption lines that can be used for a variety of applications such as environment, medical or security. We present results on broadband SLDs with spectral width > 50 nm and high spectral power density up to 1 mW/nm as well gain-chips with ultra-wide tuning bandwidth in external cavity configuration [1, 2]. Depending on the configuration, the tuning bandwidth ranges from 170 mW in Littrow configuration to > 200 nm in Littman-Metcalf with output power exceeding 4 mW in entire tuning range. Moreover, we will show recent result of hybrid external cavity laser based on GaSb gain-chip integration with SOI platform with CW output power exceeding 7 mW and tuning bandwidth of 58 nm [3].

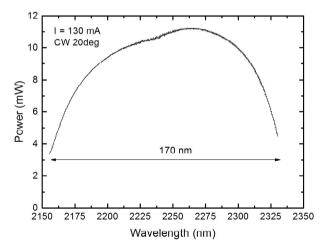


Fig. 1. Tuning bandwidth of GaSb gain-chip embedded in external cavity with Littrow configuration.

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#### **WeO10**

#### **Quantum Cascade Lasers with Nonuniformly Tapered Waveguides**

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Quantum cascade lasers (QCLs) with tapered waveguides have been reported as high-power sources of low divergent beam of mid-infrared and terahertz radiation [1-3]. Although it has been demonstrated for other types of waveguides, that a waveguide with a convex taper has better mode-conversion coupling efficiency than the one with a linear taper [4], there are no reports about QCLs with nonuniformly tapered waveguides.

In this paper we present theoretical analysis of mid-infrared QCL waveguides with linear, convex and concave tapers. We have calculated divergence and brightness of the optical beam and maximal power density on the laser front mirror for different widths and lengths of the taper. All the calculations have been performed using EigenMode Expansion Method [5] combined with the Effective Index Method.

The results of the calculations show that convex and linear tapers give beams with similar divergences much lower than concave tapers. The brightness of QCL with linear taper is better than of the one with convex or concave taper, but convex tapers give the lowest maximal power density on the mirror and are a promising option for high-power lasers.

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This work was supported by National Center for Research and Development grant Lider 317/L-5/2013.

### Triple Quantum Wells for Active Regions of Mode-Locked ICLs in the Mid-Infrared

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Recently, a dual-comb spectroscopy has been brought to the mid-infrared spectral region by means of an actively mode-locked quantum cascade laser (QCL)<sup>1</sup>, allowing for broadband field measurements with resolution of 0.0027 cm<sup>-1</sup> around 7  $\mu$ m. Although QCLs have become standard semiconductor laser sources in the mid-infrared above 4  $\mu$ m the possibility of passive mode locked operation in the saturable absorber scheme is questionable due to the very short upper state lifetime<sup>2</sup>. In contrast, interband cascade lasers (ICLs) might be considered as an alternative since they exhibit relatively long (nanoseconds) upper state lifetime<sup>3</sup>, operate in continuous wave mode up to 7  $\mu$ m at temperatures above ambient and have much lower threshold current densities than QCLs, and still produce cw output power exceeding 500 mW <sup>4</sup>. Thus, mode-locked ICLs would open a pathway for compact and low-power consuming optical sensing systems capable of detecting multiple transitions over a wide spectral range with high spectral resolution.

We present experimental verification of ICLs' active region which is better suited for passive mode-lock operation than the commonly used InAs/GaInSb W-shaped type II QW. Our approach assumes employing fast saturable absorber  $(\tau_a)$  scheme<sup>5</sup> with recovery time significantly faster than the pulse duration  $(\tau_g)$ ,  $\tau_a << \tau_g$ . To realize that the semiconductor laser is divided into two parts with a common bottom contact. As a result two sections are present, the gain and absorber, and both might be biased individually. The impact of external electric field on the oscillator strength (OS) of optical transitions will be presented. Since the assumption is to have significantly larger optical transition rates in the absorber section than in the gain section, the aim is to probe an active region design with large difference between the OS under the reversed bias (the respective carrier lifetimes are inversely proportional to the OS). Two designs based on asymmetric type II quantum wells will be compared. The first studied design utilizes a standard W-shaped band alignment, whereas the second one benefits from a triple quantum well design.

The work has been realized within iCspec project which received funding from the European Commission's Horizon 2020 Research and Innovation Programme under grant agreement No. 636930.

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### **WeO12**

### Interband Cascade Lasers on GaSb Substrates Emitting Beyond 5.6µm

### Anne Schade<sup>1</sup> and Sven Höfling<sup>1,2</sup>

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Since the first theoretical prediction by Yang [1] and the first experimental realization [2] interband cascade lasers (ICL) became more and more established, especially for gas sensing applications. The ICL combines the cascading of active stages known from the quantum cascade laser (QCL) and the interband transition of the diode lasers (DL) enabled by the semimetallic interface between InAs and GaSb. Here electrons can efficiently tunnel from the valenceband to the conduction band. The optimal workspace of ICLs is between 3-5 $\mu$ m where their low power consumption is unrivaled compared to QCLs [3] and DLs [4]. Since important gases like e.g. nitrogen oxides have strong absorption lines beyond 6  $\mu$ m, the extension beyond the actual long wavelength record of 5.6 $\mu$ m on GaSb substrates [5] is promising for many industrial applications.

Design 2 Peak Intensity Design 1 Normalized Intensity [a.u.] 1.0 Peak Intensity Design 2 [a.u.] 10 0.8 Voltage Design 1 Voltage [V] Voltage Design 2 Peak Intensity 0.6 1.0 0.4 0.5 0.2 2 0.0 5.6 5.7 5.8 5.9 6.0 6.1 0.0 0.2 0.4 0.6 0.8 1.0 1.2 1.4 1.6 1.8 2.0 2.2 2.4 2.6 Wavelength [µm] Current [A]

Fig. 1 Left: normalized spectra of broad area devices with  $100\mu m$  width and 2mm length of epitaxial structures with 4 (Design 1) and 3 (Design 2) InAs/ AlSb QW pairs in the electron injector of the active region. Right: Optical Power and Voltage in respect to the Current for broad area devices with  $100\mu m$  width and 2mm length of Design 1 and 2.

Here we discuss our recent developments of long wavelength ICLs optimized by reducing the number of InAs/ AlSb QW pairs in the electron injector of the active region. The layer design of the two represented epitaxial structures is similar except the number of QW pairs in the electron injector. Broad area devices with 100µm width and 2mm length emit at 5.72 and 5.96µm (Fig. 1 left) and show threshold current densities of 1194A/cm² of an ICL with 4 pairs of electron injector QWs and 778 A/cm² with 3 QW pairs (Fig. 1 right). The measurements were done in pulsed mode with 1kHz repetition rate and 250ns pulse duration (Fig.1). The characteristic temperature could be determined to 47K and the cut off-voltages are 1.29V and 1.33V respectively.

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# Laser Sources IV

### **Advances in Interband Cascade Lasers and LEDs**

J.R. Meyer<sup>1</sup>, M. Kim<sup>2</sup>, C.S. Kim<sup>1</sup>, W.W. Bewley<sup>1</sup>, C.D. Merritt<sup>1</sup>, C.L. Canedy<sup>1</sup>, M.V. Warren<sup>3</sup>, and I. Vurgaftman<sup>1</sup>

<sup>1</sup> Naval Research Laboratory, Washington DC 20375 <sup>2</sup> Sotera Defense Solutions, Crofton MD 21114 <sup>3</sup> ASEE Fellow Residing at NRL, Washington DC 20375

We review recent advances of interband cascade lasers (ICLs) and light emitting diodes (ICLEDs) at NRL. One investigation explored the performance of redesigned ICLs operating in the 4.6-6.1  $\mu$ m spectral range. For an ICL emitting at  $\lambda=4.8~\mu$ m, the pulsed threshold current density at room temperature was 220 A/cm², the lowest ever reported for a semiconductor laser at such a long emission wavelength. Broad-area devices emitting in the 4.6-4.9  $\mu$ m range were observed to maintain pulsed external differential quantum efficiencies (EDQEs) of 11-17% when operating at 375 K. An ICL emitting at  $\lambda=5.7~\mu$ m exhibited  $j_{th}=450~A/cm^2$  and EDQE = 27% at room temperature. Auger coefficients extracted from the recent thresholds indicate a roughly linear increase with wavelength, with the value at 6  $\mu$ m being 3-4 times higher than that at  $\lambda=3.5~\mu$ m. The Auger coefficients are also observed to increase at wavelengths shorter than  $\approx3.2~\mu$ m, for reasons that are not understood. We have also analyzed the thresholds of mid-IR diode lasers with type-I active quantum wells, for comparison to the type-II data.

Interband cascade vertical-cavity surface-emitting lasers (ICVCSELs) have operate in pulsed mode up to 70 °C [1]. The output from mesas with diameters ranging from 30 to 60  $\mu$ m (emission aperture diameters 20-50  $\mu$ m) were circularly symmetric, and the threshold current densities at T=25 °C were as low as 390 A/cm². However, the differential slope efficiencies were low ( $\leq$  50 mW/A at T=25 °C) due to loss in the top and bottom mirrors and reduced current efficiency. The smallest device operated in a single spectral mode, despite having an emission aperture much wider than the wavelength.

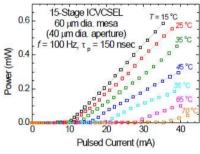


Fig. 1 - Pulsed L-I characteristics of an ICVCSEL operating at  $\lambda \approx 3.4~\mu m$ .

NRL also demonstrated improved performance for ICLEDs emitting at a peak wavelength near 3.1  $\mu m$  (FWHM  $\approx 400$  nm). The maximum cw output power of 2.9 mW, from a mesa with diameter 400  $\mu m$ , is substantially higher than the previous NRL record of 1.6 mW. The maximum wallplug efficiency of > 0.4% at low currents is also much higher than any previous result for a mid-IR LED. Since the degradation with increasing temperature is quite gradual, the maximum cw power now attainable at 105 °C exceeds the best previous result at 25 °C.

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### INVITED

### **Specialized Laser Sources for Sensing in the MIR**

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In the mid infrared (MIR) spectral region between 3 and 6 µm numerous strong absorption lines of industrial relevant gases can be found. For this reason tunable laser absorption spectroscopy (TLAS) in this wavelength window represents a key technology for the detection of very low molecule concentrations. This technology requires compact single mode laser sources with low input powers and typical optical output powers in the 1-10 mW range. These requirements can ideally be fulfilled by an interband cascade laser (ICL) [1,2], a semiconductor laser that combines radiative recombination between conduction and valence band states as in a conventional diode laser with the cascading scheme of a quantum cascade laser. This is enabled by an interband tunneling process based on the broken gap alignment between GaSb and InAs. Due to several improvements within the last decade as for example the carrier rebalancing concept [3] continuous wave (cw) operation at room temperature has been shown in the spectral range of interest.

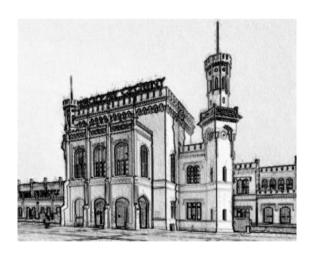
In this talk, we will report on various ICL based device concepts providing application-grade performance for high accuracy sensing application in the MIR. Corresponding gain material was grown by solid source molecular beam epitaxy (MBE) on n-GaSb wafers. Based on high quality ICL material in the MIR spectral range from 3 µm to 6 µm, single longitudinal mode selection was in general realized by the use of optimized lateral metal grating structures placed on both sides of the laser ridge using e-beam lithography [4]. These devices include e.g. DFB type devices but also mono mode device structures providing extended wavelength coverage for multi-component analysis or structures for increased output power.

The sources enable highly selective and sensitive sensors for industrial process analysis as well as for applications related to health, safety or environmental monitoring. Based on the novel laser sources various application examples are highlighted in the talk.

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<sup>&</sup>lt;sup>2</sup>Technische Physik, Physikalisches Institut and Wilhelm Conrad Röntgen Research Center for Complex Material Systems, Universität Würzburg, Am Hubland, 97074 Würzburg, Germany

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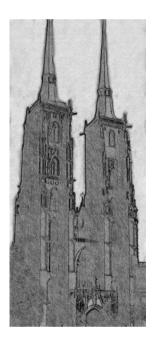


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### **Sponsors**



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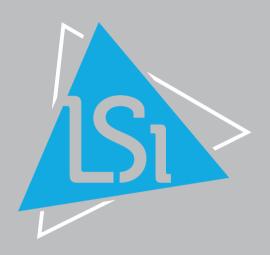
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## LASER SYSTEMS INTEGRATORS



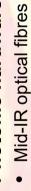
MINERYA MId- to NEaR infrared spectroscopy improved medical diagnostics for improved medical diagnostics

# www.minerva-project.eu



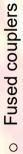






Active & passive

Mid-IR components



AO modulators 0

supercontinuum sources Ultra-long wavelength

o 1.5-4.5 µm (ZBLAN)

o 4-12 µm (chalcogenide)

o 2.9 µm

Detectors















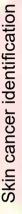
Please visit the

Calomel crystals

Novel pump lasers

T2SL technology.





Rigid probe for human skin examination

Identification of altered cells & lesions

High volume automated pathology screening Microscope-based hardware module

Rapid analysis of disease-specific chemical signatures 0

Discrimination of abnormal cells.

Gooch & Housego Gloucestershire Hospitals MFS



The University of Nottingham

EXETER









# Photonics



### NLIR provides novel uncooled ultra sensitive MIR sensors

NLIR D3055	$3.0-5.5 \mu m$	Detector
NLIR S3055	3.0-5.5µm	Spectrometer
NLIR S55100	5.5-10.0μm	Spectrometer
NLIR L3055	3.0-5.5µm	Line Spectrograph

### www.nlir.com

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