



mirs3ns

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

5 - 7 March 2015

Würzburg, Germany

Book of abstracts



**3rd International Workshop on Opportunities and
Challenges in Mid-Infrared Laser-Based Gas Sensing**

**5 – 7 March 2015
Würzburg, Germany**

Venue

Toscanasaal in the Residence of Würzburg, Würzburg, Germany

Scope

The workshop is organized by University of Würzburg and nanoplus GmbH with the support of Wrocław University of Technology. The workshop target is to provide an overview of the state of the art in the field of laser-based detection of various gases with the absorption characteristics in the mid-infrared region. At the same time, the workshop will promote and encourage interactions between academic and industrial research institutions and will address scientific and technological challenges associated with both the latest developments and future prospects. The workshop is organized as a combination of plenary lectures given by world-renowned experts and short oral contributed presentations.

The main topics of **mirs3ns** workshop:

- Semiconductor structures for the mid-infrared emission
- Electronic and optical properties of the laser active material
- Recent progress in mid-infrared laser sources
- Laser-based gas sensing and related techniques
- New application prospects



The **mirs3ns** workshop has been organized within **WideLase Project** of the 7th Framework Programme of the European Commission:
<http://www.widelase.eu/>

Committees

Organizing Committee

Martin Kamp (Chair)	<i>University of Würzburg, Germany</i>
Marc Fischer	<i>nanoplus GmbH, Germany</i>
Grzegorz Sęk	<i>Wrocław University of Technology</i>
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Jan Misiewicz	<i>Wrocław University of Technology, Poland</i>
Stefan Lundqvist	<i>Airoptic, Poland</i>

Thursday – 05.03.2015						
8:00	Reception desk open					
9:00	9:15	Opening Address				
SESSION: Laser sources I						
9:15	10:00	Manijeh Razeghi	Northwestern University	USA	Invited	Recent breakthroughs in quantum cascade lasers
10:00	10:45	Jerry Meyer	Naval Research Laboratory	USA	Invited	Interband Cascade Lasers for the Midwave Infrared
10:45	11:00	Coffee break				
SESSION: Laser sources II						
11:00	11:20	Maciej Bugajski	Institute of Electron Technology	Poland	Contributed	Low threshold, above room temperature operation of InGaAs/AlGaAs/GaAs quantum cascade lasers
11:20	11:40	Mathieu Carras	III-V Lab	France	Contributed	Advances toward monolithic widely tunable mid-infrared sources
11:40	12:00	James A. Gupta	National Research Council of Canada	Canada	Contributed	Type-I Interband Cascade Lasers Near 3.2µm
12:00	12:20	Marcin Motyka	Wroclaw University of Technology	Poland	Contributed	On the improvements in the active region of interband cascade lasers
12:20	12:40	Peter Moselund	NKT Photonics A/S	Denmark	Contributed	Progress in Mid-IR Supercontinuum
12:40	13:00	Michael von Edlinger	Nanoplus GmbH	Germany	Contributed	Monolithic widely tunable interband cascade lasers
13:00	14:00	Lunch				
SESSION: Applications I						
14:00	14:45	Frank Tittel	Rice University	USA	Invited	Compact ICL and QCL based mid-infrared sensors: development and applications
14:45	15:30	Ulrike Willer	Clausthal University of Technology	Germany	Invited	Mid-infrared photoacoustic detection schemes
15:30	16:00	Coffee break				
SESSION: Applications II						
16:00	16:20	Lorenzo Cocola	CNR Institute for Photonics and Nanotechnologies, Padova	Italy	Contributed	The FP-7 SAFETYPACK project
16:20	16:40	Peter Geiser	Norsk Elektro Optikk A/S	Norway	Contributed	Interband cascade laser based measurements of sulfur dioxide for emission monitoring applications
16:40	17:00	Michal Nikodem	Wroclaw Research Centre EIT+	Poland	Contributed	Gas sensing using photo-thermal spectroscopy with coherent signal detection
17:00	17:20	Norbert Lang	Leibniz Institute of Plasma Science and Technology	Germany	Contributed	Optical Feedback Cavity-Enhanced Absorption Spectroscopy with a 3.2 µm Interband Cascade Laser
17:20	17:40	Markus Mangold	Empa - Swiss Federal Laboratories for Materials Science and Technology	Switzerland	Contributed	Cylindrical Multipass Reflection Cells for Optical Trace Gas Sensing
17:40	18:00	Jürgen Röpcke	INP Greifswald	Germany	Contributed	Comparison of low scale and industrial scale active screen plasma nitriding processes using mid-infrared laser absorption spectroscopy
18:00		End of the day				
19:00		Conference Dinner				

Friday – 06.03.2015						
SESSION: Sources, detectors, materials I						
9:45	10:30	Antoni Rogalski	Military University of Technology	Poland	Invited	Type-II superlattice HOT infrared photodetectors
10:30	11:15	Joachim Wagner	Fraunhofer Institute for Applied Solid State Physics	Germany	Invited	Widely external-cavity tunable quantum cascade lasers for spectroscopic sensing
11:15	11:30	Coffee break				
SESSION: Applications III						
11:30	12:15	Barry McManus	Aerodyne Research Inc.	USA	Invited	Trace gas instrumentation with ICL's and QCL's, with application to field measurements
12:15	13:00	Francesco D'Amato	Aerospace Optics Group	Italy	Invited	Chemical and mechanical sensing with mid-infrared lasers
13:00	14:00	Lunch				
SESSION: Laser sources III						
14:00	14:45	Michael Santos	University of Oklahoma	USA	Invited	Recent progress in InAs-based interband cascade lasers
14:45	15:30	Aurore Vicet	University of Montpellier	France	Invited	New Index-coupled distributed-feedback GaSb-based laser diodes in the 2 to 3 μm wavelength range. Applications to spectroscopy
15:30	15:50	Coffee break				
SESSION: Sources, detectors, materials II						
15:50	16:10	Piotr Gutowski	Institute of Electron Technology	Poland	Contributed	AlInAs/InGaAs/InP quantum cascade lasers grown by combined MBE and LP-MOVPE technology
16:10	16:30	Matthias Dallner	University of Würzburg	Germany	Contributed	InAs-based Interband-Cascade-Lasers in the 6-7 μm wavelength range
16:30	16:50	Kamil Pierściński	Institute of Electron Technology	Poland	Contributed	Room temperature, single mode emission from two-section coupled cavity InGaAs/AlGaAs/GaAs quantum cascade laser
16:50	17:10	Mateusz Dykisz	Wroclaw University of Technology	Poland	Contributed	Reflectivity-based Characterization Of Doped Layers In The Infrared Device Structures
17:10	17:30	Elzbieta Machowska - Podsiadlo	Rzeszow University of Technology	Poland	Contributed	Calculations of infrared absorption in InAs/GaSb superlattices
17:30	17:50	Krzysztof Ryzko	Wroclaw University of Technology	Poland	Contributed	Novel design of type-II quantum wells for mid-IR emission with tensile –strained GaAsSb layer for confinement of holes
17:50		End of the day				

Saturday – 07.03.2015						
SESSION: Sources, detectors, materials III						
9:15	10:00	Jozef Piotrowski	VIGO System	Poland	Invited	Fast response photodetectors for mid-infrared laser-based gas sensing
10:00	10:45	Martin Kamp	University of Würzburg	Germany	Invited	High performance GaSb-based interband cascade lasers
10:45	11:00	Coffee break				
SESSION: Applications IV						
11:00	11:20	Lukas Emmenegger	Empa - Swiss Federal Laboratories for Materials Science and Technology	Switzerland	Contributed	Frontiers of QC Laser spectroscopy for high precision isotope ratio analysis of non-CO ₂ greenhouse gases
11:20	11:40	Matthias Godejohann	MG Optical Solutions GmbH	Germany	Contributed	Hyper-Spectral-Imaging Applications on the Micro scale with Quantum Cascade Lasers
11:40	12:00	Paweł Kluczyński	Airoptric	Poland	Contributed	Open path gas sensing applications in Mid-IR using the GasEye tunable laser spectrometer
12:00	12:20	Craig Richmond	Centre for Metrology and Accreditation (MIKES)	Finland	Contributed	Isotope Metrology Using Mid-IR Spectroscopy
12:20	12:40	Javis Nwaboh	Physikalisch-Technische Bundesanstalt	Germany	Contributed	Laser spectroscopic CO measurements in the near and mid infrared regions
12:40	13:00	Kavoori S. Nagapriya	GE Global Research	India	Contributed	Laser calorimetry spectroscopy for in-liquid dissolved gas detection and measurement
13:00	13:20	Béla Tuzson	Empa - Swiss Federal Laboratories for Materials Science and Technology	Switzerland	Contributed	Selective and Sensitive VOC Breath Analysis Using a 3.3 µm Broadly-Tunable VECSEL
13:20		Closing Address				
13:30	14:30	Lunch				
14:30		End of the day				

5 March (Thursday)

Sessions

Laser sources I

Laser sources II

Applications I

Applications II

INVITED

Recent breakthroughs in quantum cascade lasers**Manijeh Razeghi**

*Center for Quantum Devices, Department of Electrical Engineering and Computer Science,
Northwestern University, USA*

Thanks to the perfection of structure design, material growth, and device fabrication, quantum cascade laser has become the most sophisticated, the most versatile, and the most efficient laser source in the mid-infrared up to Terahertz. At Center for Quantum Devices of Northwestern University, we are currently holding a number of world records in terms of the output power, efficiency, operation temperature, and wavelength coverage. Our current research interest includes monolithic broadband tunability, single mode power scaling, and high power THz QCL based on difference frequency generation. In all these areas, we are continuing to lead the world in the development of high performance quantum cascade lasers.

Interband Cascade Lasers for the Midwave Infrared

Jerry R. Meyer,¹ Chadwick L. Canedy,¹ Chul Soo Kim,¹ William W. Bewley,¹
Charles D. Merritt,¹ Igor Vurgaftman,¹ and Mijin Kim²

¹ Naval Research Lab, Washington DC 20375

² Sotera Defense Solutions, Crofton MD 21114

Interband cascade lasers (ICLs) emitting in the midwave infrared have advanced dramatically in recent years, mostly due to improved designs of the active and optical confinement regions. While the more mature InP-based quantum cascade laser (QCL) has received considerable attention as a source for IR spectroscopy systems, the GaSb-based ICLs will be advantageous in most applications requiring ≤ 20 mW of single-mode output power and wavelengths in the range ≈ 3 -6 μm . This is because ICLs have lower threshold current densities and also much lower operating voltages than QCLs, making the typical drive power at least an order of magnitude smaller. Besides extending battery lifetimes, the implication is a substantial reduction of the overall footprint and weight of the source component of a fielded spectroscopic sensing system. With demonstrated drive power as low as 30 mW [1], and the potential for reduction by another factor of 3-10, one can envision using solar panels to power unattended ICL-based sensors.

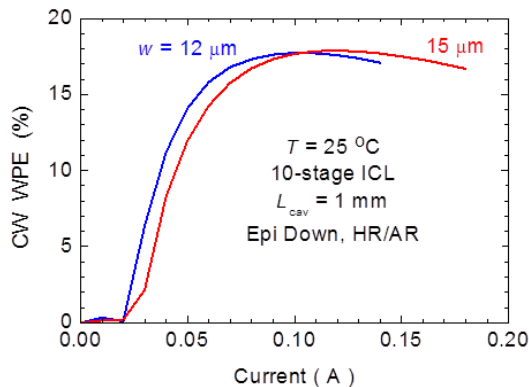


Figure 1 – CW wallplug efficiencies of 10-stage ICLs with two ridge widths.

This talk will review the current status of ICL development at NRL. Recent advances include the observation of up to 18% cw wallplug efficiency at 25 °C [2], as shown in Fig. 1, cw output power up to 464 mW with beam quality $M^2 = 1.9$, obtained by corrugating the ridge sidewalls to suppress higher-order transverse modes, and the demonstration of interband cascade LEDs whose maximum cw output power of 1.6 mW at 25 °C is higher than for any previous mid-IR LED. Limits on the minimum practical drive power and maximum wavelength tuning by a single element will be discussed.

[1] I. Vurgaftman, W. W. Bewley, C. L. Canedy, C. S. Kim, M. Kim, C. D. Merritt, J. Abell, J. R. Lindle, and J. R. Meyer, *Nature Commun.* 2, 585 (2011).

[2] M. Kim, W. W. Bewley, C. L. Canedy, C. S. Kim, C. D. Merritt, J. Abell, I. Vurgaftman, and J. R. Meyer, submitted to *Opt. Expr.*

Low threshold, above room temperature operation of InGaAs/AlGaAs/GaAs quantum cascade lasers

Maciej Bugajski¹, Piotr Gutowski¹, Andrzej Kolek², Grzegorz Haldaś²,
Kamil Pierściński¹, Dorota Pierścińska¹

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Mid-IR QCLs can be divided into two main categories based on the material system involved; GaAs- and InP-based devices. The latter offer unquestionable advantages in terms of high temperature, CW operation. The former, however, offer performance in pulsed mode operation at room temperature, which is sufficient in many applications. Besides that, the cost and complexity of technology and fabrication is much lower for GaAs based devices. The main issue that limits high temperature performance of GaAs-based devices is the low conduction band-offset, causing the electrons to escape to 3D continuum of states at elevated temperatures of the active region. There were attempts taken to increase the band-offset by e.g. increasing the Al content in the barrier layers. Unfortunately, increased Al content leads to increased inter-valley scattering to X and L minima and consequently to decreased laser performance.

In this work we report on the design, realization and characterization of a mid-IR QCLs based on InGaAs/AlGaAs/GaAs structures grown by molecular beam epitaxy. Structures were grown with indium content of 3% in QWs and 47% of Al in AlGaAs barrier layers. The design results in strained heterostructure, however, no strain relaxation was observed as documented by X-ray diffraction measurements. Devices exhibit performance largely improved over standard AlGaAs/GaAs QCLs. More than 2 times reduction of threshold current density was observed. Lasing at $\sim 9.5 \mu\text{m}$ was achieved in the pulse mode up to $T = 50^\circ\text{C}$ with characteristic temperature $T_0 = 120 \text{ K}$.

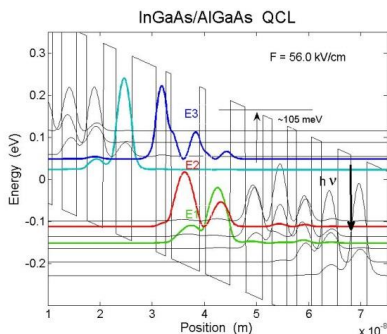


Fig.1 Conduction band profile and moduli squared wavefunctions in injector/active/injector segment of the $\text{In}_{0.03}\text{Ga}_{0.97}\text{As}/\text{Al}_{0.47}\text{Ga}_{0.53}\text{As}/\text{GaAs}$ laser under the applied field of 56 kV/cm.

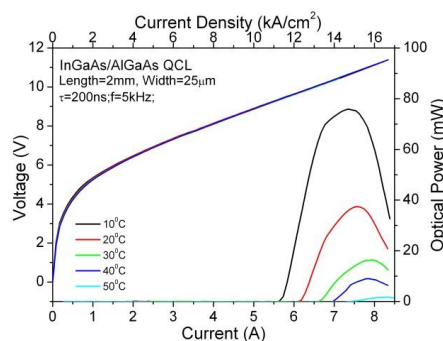


Fig.2 L-I-V characteristics of $\text{In}_{0.03}\text{Ga}_{0.97}\text{As}/\text{Al}_{0.47}\text{Ga}_{0.53}\text{As}/\text{GaAs}$ laser at high temperatures.

Nonequilibrium Green's functions model of InGaAs/AlGaAs/GaAs laser that utilizes single-phonon resonance scheme has been analyzed. This method is used to examine electronic transport, optical gain, and carrier distributions in the subbands of the laser. The problem is completely solved in k-space and nonparabolicity is accounted for through energy dependent effective mass. Scattering selfenergies included in NEGF formalism are for LO-phonon, interface roughness (IR), alloy disorder (AD) and ionized impurity (I-I) scatterings and for energy averaged LA-phonon scattering. Electron-electron scattering was included within Hartree approximation by solving Poisson equation (with the boundary conditions that preserve charge neutrality of each QCL period) selfconsistently with NEGF equations. Good agreement with experimental data is found.

This work was partially supported by PROFIT PBS 2/A3/15/2013 and by National Science Centre Grant SONATA no. 2013/09/D/ST7/03966.

Advances toward monolithic widely tunable mid-infrared sources

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P. Barritault², S. Nicoletti², L. Jorge Orbe³, G. Carpintero Del Barrio³

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3. Universidad Carlos III de Madrid, 28911 Leganes, Madrid, España

We present the last developments and results on compact, low cost, monolithic widely tuneable Mid-InfraRed (MIR) sources for spectroscopy based on arrays of Distributed Feedback (DFB) Quantum Cascade Lasers (QCL) and passive Multiplexers (MUX) on Silicon or Indium Phosphide wafers.

In previous works, we have developed a top metal grating approach to get a robust and versatile technology to realize DFB QCL [1]. This technology is very useful for the processing of DFB array since Bragg gratings are realized on the top of the waveguide. We will present last results obtained with this technology used to realize DFB QCL array in different wavelength ranges.

In order to combine all the beams from the array into a single output, two integrated solutions have been studied. The first one is based on a C-MOS compatible MIR Platform using SiGe/Si stack. Low-losses behaviour on single-mode waveguide has been measured in the 3 to 8 μm range [2]. Using this MIR Platform Array Waveguide Gratings (AWG) multiplexers working at 4.5 and 7.4 μm were designed and realized on 200 mm Si wafers, making broad band MIR passive devices affordable at low cost. The array of QCL and the AWG multiplexer are then, hybrid integrated, to form the widely tunable source [Fig. 1 a)]. The second explored technology is based on the same material stack used for QCL fabrication. InP based combiner can thus easily be monolithically integrated with the QCL array via evanescent coupling. However, since QCL are typically fabricated on 2" wafers, a more compact echelle grating configuration has been investigated instead of AWG [Fig. 1 b)].

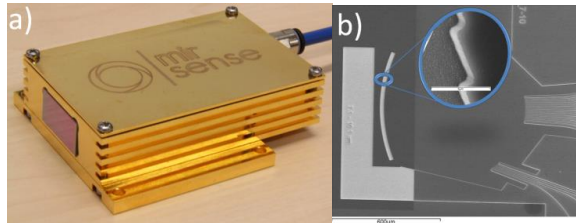


Fig. 1 a) Picture of the packaged widely tuneable MIR source based on DFB QCL array and SiGe AWG. b) SEM picture of the echelle grating at 8 μm . Inset: SEM picture, zoom on the diffraction grating tooth.

[1] M. Carras, G. Maisons, B. Simozrag, M. Garcia, O. Parillaud, J. Massies, and X. Marcadet, "Room-temperature continuous-wave metal grating distributed feedback quantum cascade lasers" Appl. Phys. Lett., 93, 011109 (2008).

[2] M. Brun, P. Labeye, G. Grand, J.-M. Hartmann, F. Boulila, M. Carras, and S. Nicoletti, "Low loss SiGe graded index waveguides for mid-IR applications" Optics Express, Vol. 22, 508 (2014).

Type-I Interband Cascade Lasers Near 3.2 μ m

**James A. Gupta¹, Geof C. Aers¹, Emmanuel Dupont¹, Jean-Marc Baribeau¹,
Xiaohua Wu¹, Yuchao Jiang², Lu Li², Rui Q. Yang² and Matthew B. Johnson³**

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The 3-4 μ m wavelength range is technologically important for mid-infrared laser-based hydrocarbon gas sensing. Several types of semiconductor lasers are being actively developed for this range, including type-I interband diode lasers [1,2] and quantum cascade lasers (QCLs) [3]. Interband cascade lasers (ICLs) have now established themselves as another very attractive solution [4]. The devices incorporate carrier recycling, as with QCLs, but the photon emission occurs via electron-hole recombination in the QW active region, as opposed to the unipolar electron intersubband recombination in conventional QCLs. Most ICL development to date has focused on type-II active regions, enabling continuous-wave (CW) operation above room-temperature with low power consumption [5].

In this work, we demonstrate the realization of type-I ICLs using InGaAsSb/AlAsSb quantum wells (QWs). The structures were grown on GaSb substrates by molecular beam epitaxy (MBE) and are based on strain-balanced InAs/AlSb superlattice cladding layers surrounding a GaSb waveguide containing the six-stage ICL active region. Each active region stage has a single In_{0.55}Ga_{0.45}As_{0.22}Sb_{0.78} QW, with an 8-QW InAs/AlSb electron injector and a 3-QW GaSb/AlSb hole injector. Broad-area lasers operated in pulsed mode up to 365K with a 300K threshold current density of $\sim 295 \text{ Acm}^{-2}$ and threshold voltage of $\sim 3.4 \text{ V}$ at a lasing wavelength of 3.13 μ m. Narrow ridge-waveguide devices operated in CW mode up to 306K. The emission wavelength is close to 3.2 μ m at 300K with a threshold current of 120mA and an output power exceeding 2mW/facet.

These preliminary results attest to the strength of the type-I ICL concept and significant improvements are expected through optimization of the design and fabrication. Ultimately, the high gain and efficient carrier injection of type-I ICLs may make these devices a technology of choice for gas sensing applications in this wavelength range.

[1] M. Grau, C. Lin, O. Dier, C. Lauer and M.-C. Amann, "Room-temperature operation of 3.26 μ m GaSb-based type-I lasers with quinary AlGaInAsSb barriers", *Appl. Phys. Lett.* **87**, 241104 (2005).

[2] T. Hosoda, G. Kipshidze, G. Tsvi, L. Shterengas and G. Belenky, "Type-I GaSb-Based Laser Diodes Operating in 3.1- to 3.3 μ m Wavelength Range", *IEEE Photonics Tech. Lett.* **22**, 718 (2010).

[3] O. Cathabard, R. Teissier, J. Devenson, J. C. Moreno, and A. N. Baranov, "Quantum cascade lasers emitting near 2.6 μ m", *Appl. Phys. Lett.* **96**, 141110 (2010).

[4] R. Q. Yang, "Interband Cascade (IC) Lasers", in *Semiconductor lasers: fundamentals and applications*, A. Baranov and E. Tournié, ed. (Woodhead Publishing Limited, Cambridge, UK, 2013), Chap. 12; and references therein.

[5] I. Vurgaftman, W. W. Bewley, C. L. Canedy, C. S. Kim, M. Kim, C. D. Merritt, J. Abell, and J. R. Meyer, "Interband cascade lasers with low threshold powers and high output powers", *IEEE J. Select. Top. Quantum Electron.* **19**, 1200210 (2013).

On the improvements in the active region of interband cascade lasers

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R. Weih², M. Dallner², M. Kamp², S. Höfling^{2,3}

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²*Technische Physik, University of Würzburg, Würzburg, Germany*

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Applications related to the detection of hazardous and environmentally-relevant gasses drive the growing demands with respect to all the sensor system components, requiring cheap and compact laser sources. This can be well fulfilled by semiconductor lasers, where one of the efficient solutions is interband cascade laser (ICL). Such devices have already been proven to emit at some wavelengths of the mid-infrared (to beyond 10 μm even), i.e. in ranges characteristic for maximal absorption of many gasses. In addition, the ICLs offer continuous wave single mode operation at room temperature between 3 and 5 μm , and significantly lower power consumption [1] than the more common quantum cascade lasers. However, ICLs still need further developments regarding especially the demanded performances at longer wavelengths, broad bandwidth or widely tunable devices.

We have investigated, both experimentally and theoretically, several modifications in the active region of the ICLs. The considered structures are based on InAs/(Ga,In)(As,Sb) materials forming a broken gap system, i.e. confining electrons and holes in spatially separated layers. The lasers are usually grown on either GaSb or InAs substrates. Our study is aimed at maximizing the optical transition oscillator strength (OS) via tailoring the electronic structure, the related strain and wave function engineering. OS is the most critical parameter of the type II system because it can allow for compensating the intrinsic losses while extending the emission wavelength or the gain bandwidth. We will cover such issues as variation of compositions and thicknesses, importance of the band offsets, the active transition intensity and external factors as temperature or electric field.

A combination of two spectroscopic techniques is used, emission-like (photoluminescence) and absorption-like (modulated reflectivity), supported by the energy level calculations employing multiband $\mathbf{k}\cdot\mathbf{p}$ theory. We demonstrate that addition of arsenic into the commonly used ternary layer of GaInSb for the holes confinement can significantly enhance the transition oscillator strength, while decreasing the overall strain and keeping still the type II design [2,3]. There is also investigated the use of a triple (or a multiple) type II quantum well structure instead of a commonly used double well “W-design”[4]. This allows for simultaneous red shift of the transitions and increase of the oscillator strength.

1. I. Vurgaftman et al., *Nature Commun.* **2**, 585 (2011)
2. F. Janiak et al., *Appl. Phys. Lett.* **100**, 231908 (2012)
3. K. Ryczko et al., *J. Appl. Phys.* **114**, 223519 (2013)
4. Motyka et al., *J. Appl. Phys.* in press (2015)

The work was performed within Project WideLase No. 318798 of the EC 7th Framework Programme.

Progress in Mid-IR Supercontinuum.

Peter M. Moselund¹, Chris Brooks¹, Lasse Leick¹

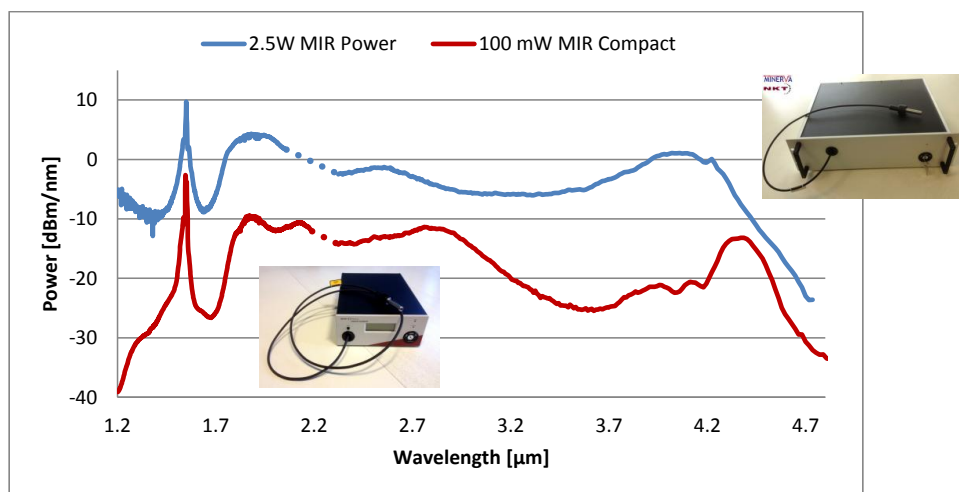
¹ NKT Photonics A/S, Blokken 84, Birkerød, Denmark

Since the first commercial broadband visible supercontinuum (SC) sources came on the market a decade ago the flexibility offered by their wide spectrum, high brightness, ruggedness and ease of use has lead to their adaptation in a wide range of imaging and sensing applications. In particularly the field of fluorescence microscopy has been revolutionized by the implementation SC systems operating in the visible domain. Now the emergence of supercontinuum sources in the mid-IR heralds a similar revolution in mid-IR sensing.

When doing spectroscopy in the Mid-IR researchers has generally had to chose between low intensity broadband thermal light sources or high intensity narrow band lasers or OPO's However Mid-IR supercontinuum is a solution which could give the best of both worlds by providing laser level intensity with lamp like spectral broadness.

In this talk we will report on the recent developments within mid-IR Supercontinuum development NKT Photonics and describe the results obtained with 1.7-4.7 μm ZBLAN ($\text{ZrF}_4\text{-BaF}_2\text{-LaF}_3\text{-AlF}_3\text{-NaF}$) based supercontinuum sources. Significantly and we prove the reliability of the source by demonstrating runtimes of over 3000 hours. The high intensity and spatial coherence these SC sources opens up a wide range of new possibilities for applications in mid-IR sensing and microscopy as we indicate by showing the road to nanosecond spectroscopy.

We will also briefly introduce MINERVA which is a currently running EU FP7 program on mid-IR cancer diagnostics using MIR supercontinuum and Mid-Tech which is an EU sponsored International Training Network for PhD students targeting mid IR lightsources and their applications in combustion sensing and bio optics.



Monolithic widely tunable interband cascade lasers

M. von Edlinger¹, R. Weih², J. Scheuermann¹, L. Nähle¹, P. Fuchs¹, L. Hildebrandt¹,
S. Höfling^{2,3}, M. Fischer¹, J. Koeth¹ and M. Kamp²

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² Technische Physik, Universität Würzburg, Am Hubland, 97074 Würzburg, Germany

³ Present address: School of Physics and Astronomy, University of St. Andrews, KY16 9SS St. Andrews, UK

Real-time, high-sensitivity gas analysis (e.g. for industrial process control) is a major field of application for monomode semiconductor lasers. Especially the mid-infrared wavelength range is of great interest for tunable laser absorption spectroscopy applications, since many technologically and industrially relevant gas species have their strongest absorption features in this spectral region. Application-grade distributed feedback (DFB) laser devices have been demonstrated in the midinfrared based on diode laser [1], quantum cascade laser [2] and interband cascade laser (ICL) [3,4] gain material.

While the current-induced tuning range of DFB lasers is usually limited to a few nanometers, there are a number of applications which benefit from lasers with wider tunability. A widely tunable laser allows to determine the base line of a measurement by sampling a spectral region far away from the broad spectral features that arise from collision broadening or large molecules, e.g. complex hydrocarbons. It can also be used for sensing of multiple gas absorption lines in a gas mixture or separating spectral features of overlapping absorption lines. A monolithic solution that is compact, rugged and provides a high tuning speed is given by a multi-segment laser device using the Vernier-tuning principle.

In this contribution, widely tunable ICL devices based on a two-section approach with binary superimposed gratings are presented. These devices use epitaxial layer structures grown by solid source molecular beam epitaxy on Te-doped GaSb substrate comparable to those described in [5]. For laser emission around 3.5 μm , designs with 5 or 6 cascades were chosen, respectively. Output powers up to 5 mW around room temperature were obtained with typical threshold currents of 50 mA. Spectral characterization was performed using a Fourier transform infrared (FTIR) spectrometer with a spectral resolution of 0.125 cm^{-1} in combination with an automated setup for continuous wave (CW) current control of the two segments. A total tuning range above 155 nm in up to six discrete wavelength channels has been achieved in CW operation around 3.7 μm . Within one channel mode hop free wavelength tuning up to 14 nm was observed. In the wavelength region around 3.3 μm , dual-channel devices for ethanol measurements in combination with corresponding base-line determination are presented. The widely tunable ICL devices promise excellent feasibility for a variety of applications, e.g. cost effective multi-gas-species analysis.

Financial support by the European Union within the FP7 project 'WideLase' (No: 318798) is gratefully acknowledged.

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Mid-infrared interband and quantum cascade laser based trace gas sensor technologies: recent advances and applications

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Recent advances of sensor systems, based on mid-infrared interband cascade lasers (ICLs) for the detection of trace gas species and their application in atmospheric chemistry, medical diagnostics, life sciences, petrochemical industry, and national security will be reported [1].

The development of compact ICL based trace gas sensors will permit the targeting of strong fundamental rotational-vibrational transitions in the mid-infrared which are one to two orders of magnitude more intense than transitions in overtone and combination bands in the near-infrared. Specifically, the spectroscopic detection and monitoring of four molecular species, methane (CH₄) [2], ethane (C₂H₆), formaldehyde (H₂CO) and hydrogen sulphide (H₂S) will be described.

CH₄, C₂H₆ and H₂CO can be detected using two innovative detection techniques: mid-infrared tunable laser absorption spectroscopy (TDLAS) using a novel, compact multi-pass gas cell and quartz enhanced photoacoustic spectroscopy (QEPAS). Both techniques utilize state-of-the-art mid-IR, continuous wave, distributed feedback ICLs and QCLs. TDLAS was performed with an ultra-compact 57.6 m effective optical path length innovative spherical multipass cell capable of 459 passes between two mirrors separated by 12.5 cm.

TDLAS and QEPAS can achieve minimum detectable absorption in the range from 10⁻⁸ to 10⁻¹¹ cm⁻¹/Hz^{1/2}. Several recent examples of real world applications of field deployable gas sensors will be described. For example, an ICL based TDLAS sensor system is capable of detecting CH₄ and C₂H₆ concentration levels of 1 ppbv in a 1 sec. sampling time., using an ultra-compact, robust sensor architecture.

H₂S detection was realized with a THz QEPAS sensor system using a custom quartz tuning fork with a new geometry and a QCL emitting at 2.913 THz [3]. The measured H₂S detection sensitivity is 30 ppm in 3 sec. and 13 ppm for a 30 sec. integration time.

Future work will include the detection of other important target analytes. Two new approaches aimed to achieve enhanced detection sensitivities with QEPAS based sensing can be realized. The first approach uses an optical power buildup cavity. The second approach will use custom-designed QTFs of different geometries. The development of cavity-enhanced optical feedback-assisted QEPAS will lead to significantly lower minimum detectable gas concentration levels of < 10 pptv.

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Mid-Infrared Photoacoustic Detection Schemes

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Basics of quartz-enhanced photoacoustic spectroscopy, several modifications of the technique and their application will be discussed.

In photoacoustic spectroscopy a modulated laser is used to selectively excite molecular species of interest. Detection is performed not by measuring an attenuation of light intensity but by sensing the acoustic wave that is formed by the periodic application of energy into the gas. An important advantage is that this technique is background free, thus the dynamic range of amplifiers can be readily used for signal enhancement. However, the generated signal depends not only on the concentration of the absorbing gas but also on molecular relaxation paths of the excited molecules. Thus, the background gas can influence signal generation and admixture of special gases can promote the de-excitation and thus enhance the signal.

The use of photoacoustic spectroscopy largely increased after introduction of quartz-enhanced photoacoustic spectroscopy (QEPAS) by Kosterev et al. [1], where a miniature quartz tuning fork (QTF) is used as a transducer instead of a conventional microphone, enabling substantial miniaturization. Due to the high quality factor of the QTF, detection limits down to the ppt range have been reached for some substances [2]. Different modifications of the technique will be discussed:

For QEPAS, the detection of the acoustic wave relies on forcing of the tuning fork into motion and the generation of a piezo current within the quartz upon bending of the tines. Since commercially available QTFs are used, the resonant frequency is fixed at 32 kHz. If a tuning fork in a different geometry is desired, e.g. for integration into a sensor chip or because of the need of a lower resonant frequency, measurement of the deflection of the tines can be performed interferometrically [3].

QTFs are commonly used as frequency standard. In this case an electric circuit is used to crop up to the resonant frequency. If such a circuit is combined with photoacoustic driving, a sensor can be realized that is automatically operated at the resonant frequency of the QTF, independently of changes in the environment. The alternate electrical and photoacoustic operation of the QTF allows also for measuring decay times instead of intensities which is of advantage if the laser intensity fluctuates.

Since the signal generated by photoacoustic spectroscopy scales linearly with incident power, usually high power lasers are used; however, with regard to the realization of miniaturized and cost efficient sensors, also the use of mid-infrared LEDs has been investigated. Besides the demonstration of LED-QEPAS, also the combination of a LED source, an absorption path and a QEPAS-based selective detector was identified as a promising sensing system [4].

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The FP-7 SAFETYPACK project

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The SAFETYPACK project, that is been funded in the framework of the FP7 EU Program and is active since November 2013, will be presented.

Aim of the project is the realization of new contactless non-intrusive laser gas sensors that will provide the food manufacturing industry with a real time inline control technology to perform quality and safety control of a wide range of sealed food.

In food packaging industry the use of gases other than air in the process of manufacturing and sealing of food items for distribution to the consumer chain (supermarkets, retail points, ...) has progressively grown. It follows that the precise measurement and control of the inside atmosphere represents a stringent requirement in the food and packaging industries. The control has to be made inline after the closure of the packaging to monitor the integrity of the seal or later to monitor the evolution of the internal gas content in time.

Inline non-intrusive contactless laser gas sensors, that are based on laser spectroscopy, will be developed and validated in the project timeframe. They will use TDLAS (Tunable diode laser absorption spectroscopy) and GASMAS (Gas in scattering media absorption spectroscopy) techniques, that will be developed and chosen tailored to the final application. The sensors will monitor the content of oxygen inside close packages and can operate both on (partially) transparent containers, such as food trays, bags, bottles of different shapes and colors, as well in almost non-transparent containers, within diffusive materials, such as paper, plastic and food itself.

The sensors will be demonstrated and validated with two real-time in-line pilot installations regarding tortilla and mozzarella cheese production.

The proposing team, coordinated by CNR-IFN Italy, consists of research institutions, universities, SMEs and industries from five different European countries. The team has already a rather strong experience in applying laser spectroscopy techniques to real-time inline control of industrial processes both in the pharmaceutical, bottling and agrofood market.

At present, the laser gas sensors for the two in-line applications have been already validated and the two machines for preliminary at-line applications are being designed.

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Interband cascade laser based measurements of sulfur dioxide for emission monitoring applications

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Nitrogen oxides (NO_x) and sulfur oxides (SO_x) are typical pollutants emitted from a vast number of all kinds of industrial processes. According to environmental protection regulations (e.g. by US EPA or Bundesumweltamt in Germany) the emission of these gases into ambient air must be limited. The increasingly strict regulations require the development of more sensitive instruments. In this presentation, a sensor for one of the most important pollutants (sulfur dioxide, SO₂) will be presented.

Tunable laser absorption spectroscopy (TLAS) has become the preferred measurement technique for many industrial applications in recent years, especially for in-situ measurements. Previously, mainly near-infrared lasers have been used in TLAS sensors. The advent of compact mid-infrared light sources like quantum cascade lasers (QCLs) and interband cascade lasers (ICLs) has made it possible to use TLAS to measure gases that do not have any absorption bands in the near-infrared, like sulfur dioxide.

Software simulations based on the HITRAN [1] database have led to a selection of interference-free SO₂ absorption lines in a typical in-situ emission monitoring application. *nanoplus* has developed a monomode DFB ICL for the 4 μm band of SO₂ [2]. The laser has been integrated in a TLAS sensor. Figure 1 shows second harmonic spectra of sulfur dioxide, water vapor, and carbon dioxide in typical concentration levels and process conditions of emission monitoring applications. The detection limit of the laboratory sensor has been measured to be less than 3 ppm·m (at ambient conditions) which corresponds to a noise level of around $2 \cdot 10^{-5}$ [rel. abs.]. Results from laboratory and field experiments will be presented.

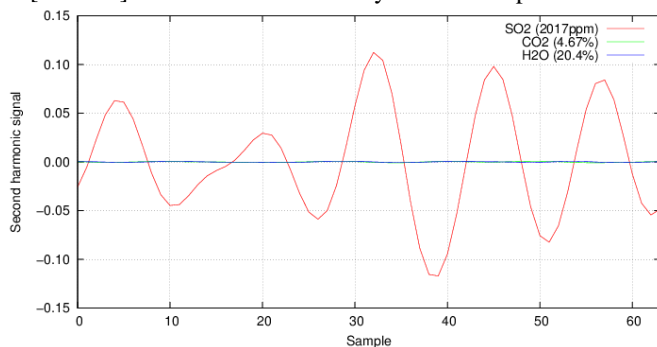


Figure 1 Second harmonic spectra of water vapor (20.4%), sulfur dioxide (2017ppm), and carbon dioxide (4.67%) in a laboratory experiment using a self-made heated cell ($T = 150\text{ }^{\circ}\text{C}$, $OPL = 70\text{ cm}$).

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Gas sensing using photo-thermal spectroscopy with coherent signal detection

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We present results on molecular detection based on absorption-induced photo-thermal effects in gases. This approach to laser gas spectroscopy is to a certain degree similar to the photo-acoustic technique. Photo-thermal effect is observed when gas molecules are being excited using coherent radiation with frequency matching the molecular resonance. The molecules subsequently relax to the ground state via non-radiative processes or emission of photons. The whole process results in a local temperature rise in the sampled air and, eventually, in the change of the gas refractive index (causing changes of the effective optical path length). Detecting these changes can be used to retrieve the information on the target analyte concentration. The proposed sensing approach can be used in any wavelength region, and is especially suitable to perform measurements in the mid-infrared where high power quantum- and interband-cascade lasers are available and the strongest fundamental ro-vibrational transitions can be targeted. While mid-IR can be used for ‘pumping’ the molecules, high quality short wavelength laser source can be used to precisely detect optical path-length variations associated with photo-thermal effects.

Proof of concept experiments were conducted in the near-IR using hydrogen cyanide (HCN) as the target gas (15 cm long cell, pure HCN at 10 Torr, with ~40% peak absorption for transitions in the 1550 nm region). Schematic diagram of the setup is shown in Figure 1. The detection was performed in a laser-Doppler vibrometry (LDV)-like setup which is a Mach-Zehnder interferometer with a gas cell in one arm and a frequency shifter (AOM, driven at $\Omega = 40$ MHz) in the other. A heterodyne beatnote between the two light waves is detected by the photodetector and changes in the optical length of either arm result in variations of the beatnote frequency. Approximately 200 mW of optical power was available at the output of the EYDFA (erbium/ytterbium doped fiber amplifier) used for photo-thermal pumping. The pump laser was intensity-modulated with a mechanical chopped at frequency $f_m \approx 600$ Hz.

Example raw signals are presented in Figure 1 where FM demodulated beatnote signal is shown for two wavelengths of the pump laser (on- and off-line). When the DFB 1 laser was tuned to match the center of HCN absorption line (P8 in this particular case) a train of clear spikes with period matching the chopping frequency was observed. These spikes are not presented in the off-line measurement (the residual signal visible in this time trace is wavelength independent and originates from heating/cooling of the optical fiber and band-pass filter that were used in-line with both the pump and the probe laser). The observed signals clearly exhibit absorption-induced changes in the optical length of the gas cell.

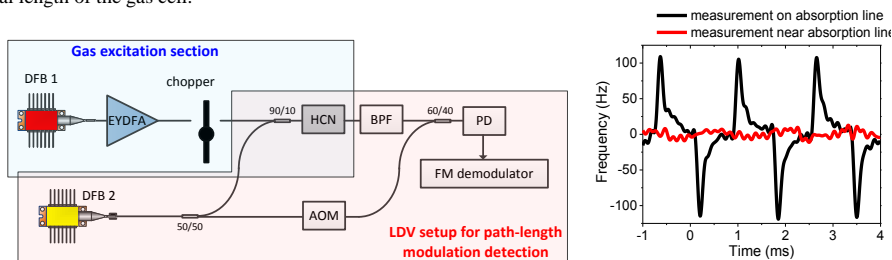


Figure 1. Left: DFB – distributed feedback laser diode, X0/Y0 – fiber couplers with appropriate split ratio, BPF – band pass filter for blocking DFB 1, PD – photodiode. DFB 1 with EYDFA and chopper were used to excite HCN molecules; DFB 2, AOM and fiber couplers formed a heterodyne Mach-Zehnder interferometer used to detect changes in the optical path length inside the gas cell (these changes affect the frequency of the beatnote at Ω). Right: FM demodulated beatnote signals indicate changes in optical path when DFB 1 is tuned to the center of the HCN transition, while no photo-thermal signal is observed when DFB 1 is tuned away from the transition.

During the conference we will present our most recent results (including studies on the dynamics of the gas heating/cooling process) and introduce an optimized detection scheme. A configuration for remote, path-integrated detection (a standoff photo-acoustic spectroscopy approach) as well as potential implementations of mid-IR QCLs or ICLs will be discussed.

Acknowledgments: MN acknowledges support from Wrocław Research Centre EIT+ within the project "The Application of Nanotechnology in Advanced Materials" - NanoMat (POIG01.01.02-02-002/08) financed by the European Regional Development Fund (Operational Programme Innovative Economy, 1.1.2). GW acknowledges support by the NSF ERC MIRTHE (EEC-0540832). KK acknowledges support from the National Science Centre within the project DEC-2012/07/B/ST7/01476 and FNP Start program.

Optical Feedback Cavity-Enhanced Absorption Spectroscopy with a 3.2 μm Interband Cascade Laser

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Interband cascade lasers (ICLs) open up the possibility to probe the strong C-H transitions in the 3 μm spectral region. We present the first, to our knowledge, demonstration of a single mode 3.24 μm distributed feedback ICL (nanoplus GmbH) coupled to a V-shaped optical cavity in an optical feedback cavity-enhanced absorption spectroscopy (OF-CEAS) experiment. The ICL clearly demonstrated feedback locking to the resonant V-shaped cavity, and showed long-term phase stability indicating some degree of self-locking. A typical example of the cavity transmission for 189 ppb CH_4 in buffer gas at a total pressure of 190 mbar is presented in fig. 1. We achieved a minimum detectable sensitivity α_{min} of $1.3 \times 10^{-7} \text{ cm}^{-1}$ for a spectrum of CH_4 with a two second acquisition time (100 scans averaged) which corresponds to a detection limit of 3 ppb at atmospheric pressure. Despite the relatively low finesse of 1900, this demonstrates that OF-CEAS with an ICL probing the strong C-H transitions in the 3 μm spectral region has a similar sensitivity for CH_4 detection as reported OF-CEAS instruments with diode lasers or quantum cascade lasers [1, 2]. The ability to frequency lock an ICL source in the important 3 μm region to an optical cavity holds a great deal of promise for future spectroscopy.

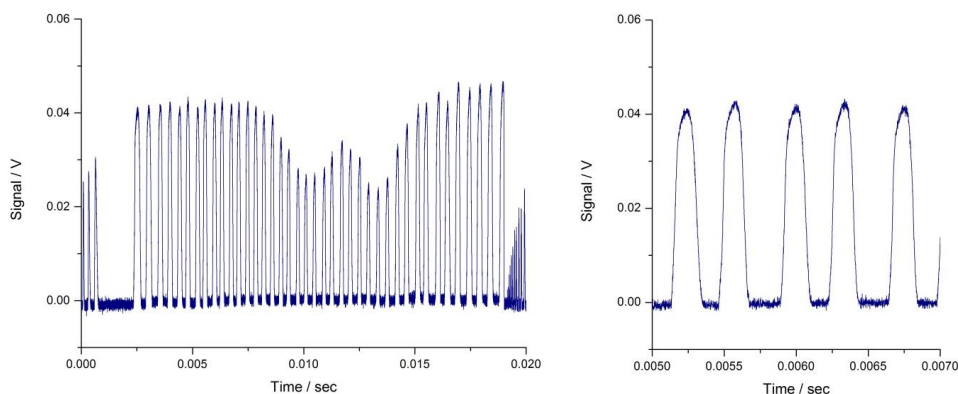


Fig. 1: Left – cavity transmission for 189 ppb CH_4 in buffer gas at a total pressure of 190 mbar while the laser is scanning to lower wavenumber. This transmission spectrum is an average of 100 scans with controlling the phase-locking. Right – a magnification of the modes around 6 ms.

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Cylindrical Multipass Reflection Cells for Optical Trace Gas Sensing

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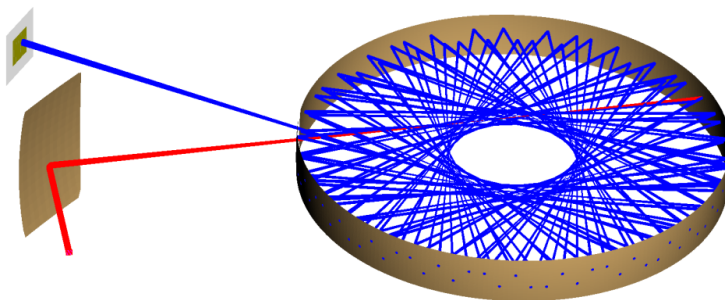
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The optical path length through a gas sample is a decisive parameter in laser spectroscopy to achieve high instrumental sensitivity and analytical precision. Small absorption signals, either due to a low number density of molecules in gas samples or weak absorption line strengths, are usually compensated for by the use of multipass cells or cavity-enhanced methods.

Recently, we have introduced a new design of a gas cell employed for MIR laser spectroscopy [1]. It consists of a single-piece, diamond turned 80 mm diameter copper cylinder. In the plane of the light beam, a toroidal mirror is carved into the cylinder surface. Due to the concentric arrangement of the toroidal surface, the light beam is refocalized after each reflection. This leads to minimal aberration and reproducible propagation of the laser beam. In this configuration, we achieved an optical path of 2.3 m in a sample volume of less than 40 ml [2].

To further improve the path to volume ratio, we have evaluated new optical configurations for such single-piece cylindrical gas cells. The considerations included different mirror shapes such as spherical and parabolic, the comparison of concentric and confocal arrangements as well as the effect of new star-patterns on the stability of the optical alignment. As a result, we have developed a multipass cell, where a parabolic shape in a confocal mirror arrangement is carved into the cylinder wall. The parabolic mirror allows for new optical configurations, e.g. an off-axis beam pattern as shown in the simulation below. Off-axis beam patterns increase the distance between adjacent reflections and thereby minimize the overlap of different optical paths.

Finally, we present a thorough analysis of the shape-accuracy that is required for the successful production of cylindrical multipass cells. We have found that while some parameters are very tolerant with respect to distortions, others have very narrow allowances. This knowledge facilitates the evaluation of new fabrication technologies allowing for low-cost mass-production of cylindrical multipass cells. By this, we aim at making one-piece cylindrical gas cells available for the fast growing mid-infrared gas sensing market.



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Comparison of low scale and industrial scale active screen plasma nitriding processes using mid-infrared laser absorption spectroscopy

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The active screen plasma nitriding (ASPN) is an advanced technology for the nitriding of steel components which has been proven its industrial applicability [1]. However, the treatment processes and, in particular, plasma chemical phenomena are far from being fully understood [2]. For this purpose a special designed compact plasma nitriding monitoring reactor (PLANIMOR), providing an independent variation of the geometry and the discharge parameters, has been constructed and is compared with an industrial scale ASPN reactor.

This study presents the results of spectroscopic investigations of pulsed DC plasma processes containing H_2 and N_2 with admixtures of CH_4 in PLANIMOR, a reactor with an inner reaction volume of about 2 dm^3 and a linear configuration of the electrode setup, and in an industrial scale ASPN reactor of 1 m^3 size.

Using *in-situ* mid-infrared absorption spectroscopy the concentration of the precursor, CH_4 , and of the reaction products, NH_3 and HCN could be determined. As radiation sources an EC-QCL (Daylight Solutions) (QCLAS) and tunable diode lasers (TDLAS) have been used in PLANIMOR and in the ASPN reactor, respectively. Using optical emission spectroscopy (OES) the trends of atomic hydrogen, molecular nitrogen and of the nitrogen ion emissions were qualitatively monitored.

Fig. 1 shows the dependencies of the concentration of the detected species monitored by TDLAS and QCLAS depending on the plasma power of the active screen comparing the industrial ASPN reactor and PLANIMOR.

Furthermore other dependencies, i.e. the gas mixture, the total pressure and the partial pressure of the precursor, were in the focus of this comparative study.

Using the (0-0) band of the first negative system of the N_2^+ -ion recorded by OES simultaneously, the gas temperature in the plasma was found to be up to 850 K and 950 K for PLANIMOR and the ASPN reactor, respectively.

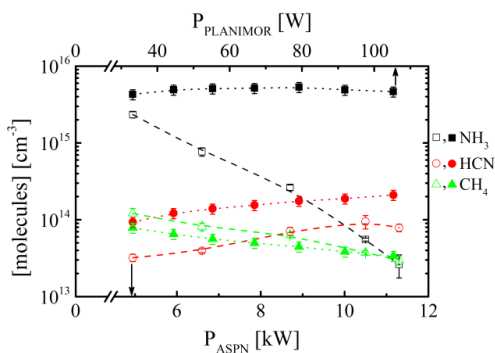


Fig. 1: The concentration of the detected species depending on the plasma power measured by TDLAS in the industrial ASPN reactor (open symbols) and by QCLAS in PLANIMOR (solid symbols) ($p = 3\text{ mbar}$; $H_2:N_2 = 1:1$, $CH_4 = 1\%$).

Comparing PLANIMOR with the industrial scale ASPN reactor, the investigated process parameters show similar influences on the monitored molecular species and on the emission properties of the plasma of the active screen.

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6 March (Friday)

Sessions

Sources, detectors, materials I

Applications III

Laser sources III

Sources, detectors, materials II

Type-II superlattice HOT infrared photodetectors

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Recently, a new strategy to achieve high-operating temperature (HOT) infrared photodetectors including barrier and cascade devices has been observed. Another method to reduce dark current is related to the limitation of the volume of detector material via a concept of a photon-trapping detector.

The paper presents approaches, materials, and device structures of the new types of infrared detectors. The intent is to concentrate on device approaches that are having the most impact today in the main stream of infrared detector technologies. A secondary aim is to outline the evolution of detector technologies showing why certain device designs and architecture have emerged as more useful today also as alternative technologies competitive to HgCdTe ternary alloy.

The performance of an innovative HOT detector designing so-called interband (IB) cascade type-II InAs/GaSb superlattice detectors is presented. Detailed analysis of the detector's performance (such as dark current, RA product, current responsivity, and response time) versus bias voltage and operating temperatures (220 to 400 K) is performed, pointing out the optimal working conditions.

The performance of nBn detector and cascade detector is compared with HgCdTe HOT detectors. At the present stage of technology, the experimentally measured R_0A values of the IB cascade type-II superlattice detectors at room temperature are higher than those predicted for HgCdTe photodiodes. It is shown that these HOT detectors have emerged as the competitors of HgCdTe photodetectors.

External-cavity Quantum Cascade Lasers for Spectroscopic Sensing

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Continuous tuning of quantum cascade lasers (QCL) over a wide wavelength range can be achieved by placing a QCL chip with a broad gain spectrum into an external cavity (EC-QCL), employing e.g. a diffractive grating as wavelength-dependent feedback-element. This way wavelength tunability over >25% of the central wavelength can be achieved routinely in the mid-infrared (MIR) spectral range, which is of particular interest for molecular finger print spectroscopy. EC-QCL technology offers further the advantage of a high spectral brightness combined with a collimated low-divergence output beam, this way enabling a range of new applications for MIR spectroscopy e.g. in non-contact chemical analysis of substances as well as in stand-off and sensing.

Here we report on recent advances in broadband-tunable MIR EC-QCL technology as well as their use in spectroscopic process analysis and imaging stand-off detection of hazardous substances. First results are presented on rapid scan EC-QCL, employing a custom-made MOEMS scanning grating in Littrow-configuration as wavelength-selective optical feedback element (Fig. 1). This way, a scanning rate of 1 kHz was achieved, which corresponds to 2000 full wavelength scans per second. Furthermore, exemplary case studies of EC-QCL based MIR spectroscopy will be presented. These include time-resolved analysis of catalytic reactions in chemical process control, as well as imaging backscattering spectroscopy (Fig. 2) for the detection of residues of explosives and related precursors in a relevant environment.



Figure 1: Photograph of an EC-QCL module employing a MOEMS scanning grating in Littrow-configuration as wavelength-selective optical feedback element.



Figure 2: Stand-off detection system based on imaging MIR backscattering spectroscopy, using EC-QCLs for wavelength-selective illumination of the scene to be investigated.

Trace gas instrumentation with ICL's and QCL's, with application to field measurements

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The recent development of commercially available inter-band cascade lasers (ICL's), combined with similarly available quantum cascade lasers (QCL's) offers new choices for trace gas instrument designers, and closes the gap in convenient choices for coherent, monolithic infrared laser sources in the spectral region with the strongest molecular absorptions. In particular, commercially available ICL's operating near 3 μm wavelength helps to provide facile access to the spectral region with the strongest absorption for molecules with C-H stretch excitations, especially many hydrocarbons. Aerodyne Research, Inc. (ARI) instruments designed for QCL's [1], have recently employed ICL's, with comparable high precision results. To accommodate the ICL's, small modifications to our basic instrument design are needed. As with any instrument with multipass cells based on high reflectivity mirrors and operating near 3 μm , the absorption by water absorbed within the coatings is a concern, and custom coatings are needed. We have found that we can routinely measure trace gas absorption with ICL's and QCL's at the absorption noise level of $\sim 5\text{e-}6$, which yields concentration noise of ~ 10 ppt (parts per trillion) in some cases.

In this paper we present details on the operation of an instrument configured for the measurement of ethane and methane, using an ICL with emission at $\sim 2997\text{ cm}^{-1}$. The instrument is built into a rack-mountable box (measuring 50 x 70 x 25 cm), containing both optics and electronics. There are additional elements to the instrument system that are external to the optics-electronics box, the air sampling pump and a circulating water chiller. The water chiller removes heat from the base of the laser and maintains the temperature of the optics enclosure. In this instrument the quantitative spectroscopic measurement of trace gases is based on direct absorption (76 m absorption path length) with spectral averaging and quantitative fitting. Proprietary computer software (TDL-WINTEL) controls the instrument and processes the data to give real time gas concentrations, at data rates up to 10 Hz. The ethane sensitivity for this instrument is typically ~ 20 ppt with 1s averaging. The good sensitivity of the instrument can be maintained while in motion in a truck.

The ethane instrument has been combined with a suite of other atmospheric instruments in Aerodyne's Mobile Laboratory and it has been used in field experiments. The main use of ethane measurements in our field experiments is to distinguish sources of methane, when quantifying methane emission fluxes. Natural gas usually has a few percent ethane in addition to the majority methane. Methane emissions from biological sources (e.g. livestock and wetlands) usually have no associated ethane. Methane emissions from natural gas facilities may be measured with tracer techniques, wherein another gas (the tracer) is released at a known rate at the facility, and then the methane and tracer plumes are measured downwind, in cross-plume traverses of the mobile laboratory [2]. Simultaneous detection of an ethane plume ensures that the methane source is in the natural gas facility.

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[2] T. I. Yacovitch, et al., *Environ. Sci. Technol.* **48**, 8028–8034 (2014).

Chemical and Mechanical Sensing with Mid-infrared Lasers

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Mid-infrared lasers are commonly used for spectroscopic applications, aiming to the quantitative detection of molecular species. The range of these applications, which started with the introduction of lead salts lasers, has significantly improved with the development of new sources. If we limit our survey to monolithic devices, now room temperature DFB diodes, ICSs and QCLs are available, so that a wide variety of measurements can be performed.

This contribution will span from an "usual" measurement, which is going to exploit the evolution of the sources (a); a new isotopic measurement, for which the new sources are crucial (b); a new application, for which external cavity QCLs compete with CO₂ laser (c).

(a) In the last 20 years mid infrared lasers evolved from lead salts lasers to quantum cascade lasers (QCLs) and interband cascade lasers (ICLs). The COLD instrument [1] has measured CO on board of a stratospheric platform (M55 Geophysica), using a lead salts laser. Now it is going to exploit this evolution, together with the evolution of the commercial electronics for these applications. The project of the device will be shown, with a discussion about the choice between QCL and ICL, both available at the required wavelength.

(b) Many analyzers, both academic and commercial, measure the isotopic ratio of carbon (in CO₂ or CH₄) or nitrogen (in NH₃), or oxygen/hydrogen (in H₂O). No one measures the isotopic ratio of chlorine in HCl. Yet, there is an interest in this matter, raising from geochemical issues. This isotopic ratio in geological emissions is not a worldwide constant. A correlation between this ratio and several characteristics of volcanic plumes could yield useful informations of the status of the volcan and, maybe, of its temporal proximity to eruption. The first results from this analyzer will be described.

(c) There are other kinds of measurements that can be carried out in the mid-infrared. Holography was limited to the visible region just because of lack of suitable detectors, as a useful source (e.g. the CO₂ laser) is available since several decades. Now thermographic cameras can record holograms, and infrared digital holography has been demonstrated, for instance for safety applications [2]. Yet, CO₂ laser is bulky, and requires high voltages. A QCL could be used instead [3]. This kind of source is less powerful, thus reducing the dimensions of the scene to be monitored. But its tunability can be exploited to obtain phase images at different wavelengths, which can be compared in order to get phase unwrapped images at synthetic, much longer wavelengths.

[1] S. Viciani, F. D'Amato, P. Mazzinghi, F. Castagnoli, G. Toci, and P.W. Werle, *Appl. Phys. B* **90**, 581 (2008)

[2] M. Locatelli, E. Pugliese, M. Paturzo, V. Bianco, A. Finizio, A. Pelagotti, P. Poggi, L. Miccio, R. Meucci, and P. Ferraro, *Optics Express* **21**, 5379 (2013)

[3] M. Ravarò, M. Locatelli, E. Pugliese, I. Di Leo, M. Siciliani de Cumis, F. D'Amato, P. Poggi, L. Consolino, R. Meucci, P. Ferraro, and P. De Natale, *Opt. Lett.* **39**, 4843 (2014)

Recent Progress in InAs-based Interband Cascade Lasers

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Interband cascade (IC) lasers [1] take advantage of the broken band-gap alignment in type-II quantum wells to reuse injected electrons in cascade stages for photon generation with high quantum efficiency. The threshold current density and power consumption can be very low because the lifetime is long for interband transitions. Much of the research on IC lasers has focused on the 3 to 4 μm region, with AlSb/InAs superlattice (SL) cladding layers and growth on GaSb substrates. Currently, IC lasers can operate in continuous wave mode at room temperature and above for wavelengths from 2.9 to 5.7 μm [2].

We present our recent progress in IC laser structures grown on InAs substrates, which use plasmon cladding layers instead of SL cladding layers [2]. This improves thermal dissipation and widens the choices of waveguide configurations. Recently, InAs-based IC lasers at wavelengths longer than 11 μm have been demonstrated as shown in Figure 1. This validated the capability of InAs-based IC lasers to cover a wide mid-infrared spectrum.

More recently, the device performance of InAs-based IC lasers has been significantly improved. For example, the threshold current density was reduced to about 340 A/cm² for broad-area devices at 300 K with a lasing wavelength near 5.2 μm . These devices can be operated in pulsed mode at up to 360 K as shown in Fig. 2. The threshold current density is the lowest ever achieved among mid-infrared lasers at this wavelength. It should be possible to achieve cw operation at room temperature when narrow-ridge devices are made from the same wafer with good thermal management. Devices made from another recent wafer at longer wavelengths (near 6.4 μm) also exhibited improved performance with a threshold current density below 1 kA/cm² at 300 K. Updated results will be reported at the workshop.

This work was partially supported by the NSF (ECCS-1002202 and IIP-1346307) and by C-SPIN, the Oklahoma/Arkansas MRSEC (NSF DMR-0520550).

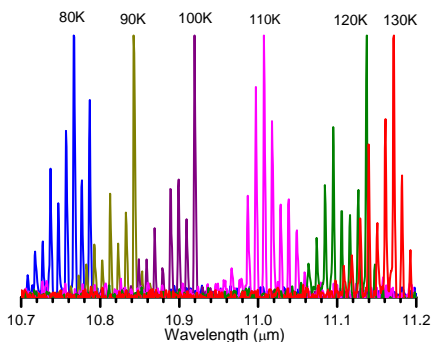


Fig. 1. Pulsed lasing spectra of broad-area lasers at temperatures of 80 to 130 K.

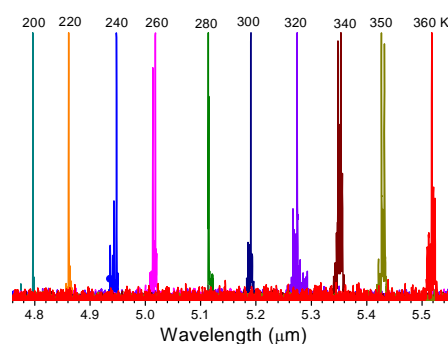


Fig. 2. Pulsed lasing spectra of 100 $\mu\text{m} \times 1.5$ mm lasers at 200 to 360 K.

[1] R. Q. Yang, Superlattices and Microstructures **17**, 77 (1995).

[2] R. Q. Yang, Chap. 12 in *Semiconductor lasers: fundamentals and applications*, edited by A. Baranov and E. Tournie (Woodhead Publishing Limited, Cambridge, UK), 2013.

New Index-Coupled Distributed-Feedback Gasb-Based Lasers Diodes in the 2 to 3 μ m Wavelength Range. Applications to Spectroscopy

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Tunable single-frequency lasers in the 2.3 to 3 μ m wavelength range, working in continuous wave regime, at room temperature, are needed to develop trace gas sensors, to identify and quantify several gases such as methane and ethylene for environmental purposes. We report on the design and development of 2nd order distributed-feedback (DFB) antimonide-lasers diodes working in this wavelength range.

The structures were grown by molecular beam epitaxy on GaSb substrate. We have chosen an index-coupled approach of the Bragg filter, in order to not degrade the lasers performances. We performed simulation analysis to adjust the Bragg grating period and the global geometry of the structure, to optimize both modal discrimination and optical power of the lasing mode. The grating is realized by holographic lithography. 2 technological processes were investigated:

- Side-wall corrugation of the laser ridge, based on an Inductively Coupled Plasma Cl-based deep etching [1,2].

- Buried grating, based on an epitaxial regrowth procedure [3].

We will present the performances of the components working at various wavelengths from 2.2 to 3 μ m (figure 1), realized from both technological developments. They exhibit side mode suppression ratio (SMSR) up to 30dB and the optical power reaches 30mW on a few nanometer large continuous tuning range. The application of these devices on spectroscopy will be shown on methane detection.

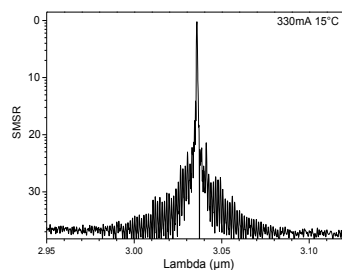


Figure 1. Emission spectrum of a 3 μ m DFB epi-regrown laser.

This work is supported by the ANR NexCILAS international project, ANR MIDAS project, by NUMEV labex and RENATECH technological support.

[1] S. Forouhar, R.M. Briggs, C. Frez, K.J. Franz and A. Ksendzov. *Appl. Phys. Lett.* 100, 031107 (2012)

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[3] Q. Gaimard, L. Cerutti, R. Teissier, A. Vicet, *Appl. Phys. Lett.* **104**, 161111-1-161111-4 (2014)

AlInAs/InGaAs/InP quantum cascade lasers grown by combined MBE and LP-MOVPE technology

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In this paper we report on hybrid approach to manufacturing InP based mid-IR QCLs. MBE grown AlInAs/InGaAs active region of the laser was combined with LP-MOVPE grown thick InP waveguide layers. Growth of the active region was performed by solid source MBE on Riber Compact 21T reactor. Epitaxial overgrowth was performed using AIXTRON 3x2" FT LP-MOVPE setup with vertical, shower-head reactor. The high resolution X-ray diffraction (HRXRD) was used to control active region quality before and after MOVPE regrowth.

The lattice-matched AlInAs/InGaAs/InP (9.2 μm) quantum cascade lasers were grown. The cross section of the laser structure is shown in Fig. 1. Bottom waveguide as well as an active region (core of the laser), marked as green and grey layers, were grown by MBE. The waveguide from the bottom side was formed by a low doped InP substrate on which 500 nm layer of lightly-doped InGaAs was grown. Active region of the laser was designed as 4-well 2-phonon resonance structure, with sequence of the layers: **4.0**, 1.9, **0.7**, 5.8, **0.9**, 5.7, **0.9**, 5.0, **2.2**, 3.4, **1.4**, 3.3, **1.3**, 3.2, **1.5**, 3.1, **1.9**, 3.0, 2.3, 2.9, 2.5, 2.9 nm. Bolded layers stand for AlInAs, whereas underlined were n doped to $2.0 \times 10^{11} \text{cm}^{-2}$. As a final MBE layer of the structure, before its transfer to the LP-MOVPE reactor, the 500 nm of lightly-doped InGaAs was grown. Then, the process was completed by LP-MOVPE growth of InP-based top waveguide consisting of two 1.5 μm thick layers; low doped ($n=4 \times 10^{16} \text{cm}^{-3}$) and higher Si-doped ($n=1 \times 10^{17} \text{cm}^{-3}$) n-type layers covered by a heavily ($n=6 \times 10^{18} \text{cm}^{-3}$) Si-doped contact layer.

500 nm	InP	$n=6 \times 10^{18} \text{cm}^{-3}$	Top waveguide (LP-MOVPE)
1.5 μm	InP	$n=1 \times 10^{17} \text{cm}^{-3}$	
1.5 μm	InP	$n=4 \times 10^{16} \text{cm}^{-3}$	
500 nm	InGaAs	$n=4 \times 10^{16} \text{cm}^{-3}$	(MBE)
30 x AlInAs/InGaAs ?			Active region (MBE)
500 nm	InGaAs	$n=4 \times 10^{16} \text{cm}^{-3}$	Bottom waveguide
500 μm substrate	InP	$n=2 \times 10^{17} \text{cm}^{-3}$	

Fig.1 Layer structure of AlInAs/InGaAs/InP lasers

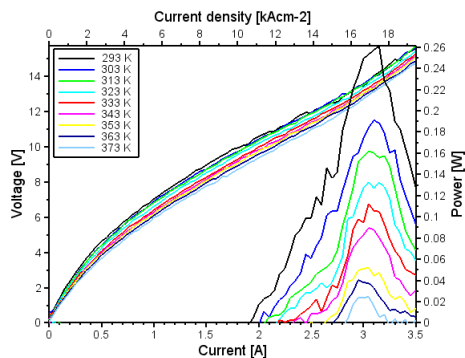


Fig.2 Light-current and current-voltage characteristics of the $\text{Al}_{0.48}\text{In}_{0.52}\text{As}/\text{In}_{0.53}\text{Ga}_{0.47}\text{As}/\text{InP}$ ($\lambda = 9.2 \mu\text{m}$) laser driven by 200 ns pulses with repetition 1 kHz at different ambient temperatures.

The double trench lasers were fabricated using standard processing technology, i.e., wet etching and Si_3N_4 for electrical insulation. The low resistivity, RTA alloyed at 370°C for 60s, Ti/Au ohmic contacts to epi-side and AuGe/Ni/Au at the substrate side of the device were used. For current injection, windows were opened through the insulator with width 15 μm and 25 μm . The lasers were cleaved into bars of 3 mm long and soldered epi-side down to Au-plated copper mounts. We have used an indium soldering or a direct Au/Au bonding. The device mounting technique has been optimized for efficient heat management. The room-temperature light-current and current-voltage characteristics of the laser are shown in Fig.2.

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InAs-based Interband-Cascade-Lasers in the 6-7 μm wavelength range

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The mid-infrared spectral region features several prominent vibrational-rotational modes of important industry gases, thus creating the need for matching reliable laser sources with low power consumption. Following the vast improvements achieved in the development of interband cascade lasers (ICLs) in recent years, ICLs are now able to meet the demands coming from applications such as tunable laser absorption spectroscopy (TLAS) over a broad spectral range. Emission wavelengths up to 5.6 μm can already be covered in cw operation at room temperature by GaSb-based ICLs [1, 2]. At longer wavelengths, ICLs grown on InAs-substrates are typically used, which utilize plasmon enhanced claddings made of highly doped InAs layers. Here emission wavelengths up to 6.1 μm [3] and 10.4 μm [4] have been achieved in pulsed operation at room temperature and cryogenic temperatures, respectively.

In this contribution we report on the design, fabrication and characterization of InAs-based ICLs operating at room temperature with an emission wavelength in the 6-7 μm range. To optimize the mode guiding for lasers emitting around 6 μm , a variation of the thickness of the InAs waveguide layer from 750 nm to 1550 nm was investigated. The lowest threshold current density of 1.2 kA/cm^2 was found for a waveguide thickness of 1350 nm at a temperature of 20 $^{\circ}\text{C}$. For devices emitting around 7 μm , the number of stages was increased to account for the higher losses at longer wavelengths. At 20 $^{\circ}\text{C}$ a deeply etched 2 mm x 45 μm device with 22 cascades exhibited a threshold current density slightly below 1 kA/cm^2 (see. Figure 1) under pulsed operation. A maximum operation temperature of 55 $^{\circ}\text{C}$ was achieved for these lasers.

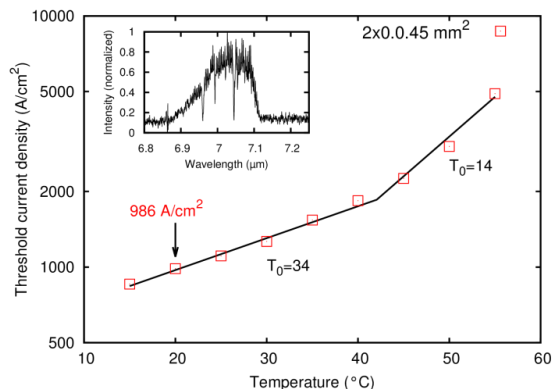


Fig. 1: Threshold current densities versus temperature for a 2 mm x 45 μm InAs ICL with 22 cascades. An emission spectrum of the device at 20 $^{\circ}\text{C}$ is shown in the inset.

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- [2] W. W. Bewley, C. L. Canedy, C. S. Kim, M. Kim, C. D. Merritt, J. Abell, I. Vurgaftman, and J. R. Meyer, *Opt. Express* **20** (3), 3235-3240 (2012).
- [3] M. Dallner, S. Höfling and M. Kamp, *Electron. Lett.*, **49**, 4, (2013).
- [4] Z. Tian, L. Li, H. Ye, R.Q. Yang, T.D. Mishima, M.B. Santos, and M.B. Johnson, *Electron. Lett.*, **48**, 2, 2012.

Room temperature, single mode emission from two-section coupled cavity InGaAs/AlGaAs/GaAs quantum cascade laser

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Single mode lasers with high side-mode suppression ratio (SMSR) and narrow linewidth are of great interest for chemical sensing applications. This can be achieved by cutting a laser cavity into 2 non-equal electrically isolated sections with an air gap in between. In this paper, room temperature, single mode, pulsed emission from two-section coupled-cavity (TS CC) InGaAs/AlGaAs/GaAs quantum cascade laser (QCL) is demonstrated and analyzed. Focused ion beam (FIB) processing has been used to fabricate investigated devices (see Fig.1).

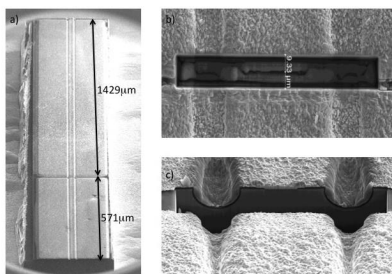


Fig.1 SEM image of FIB processed TS CC InGaAs/AlGaAs/GaAs laser

The single mode emission wavelength is centered at 1058.4 cm^{-1} ($9.45\mu\text{m}$). Side mode suppression ratio of 30dB was achieved on applying 1.6A current to the short section (300ns/5kHz) and 8.9A current to the long section (100ns/5kHz) (Fig.2). The laser exhibits a peak output power in the range of 10 mW per facet at room temperature.

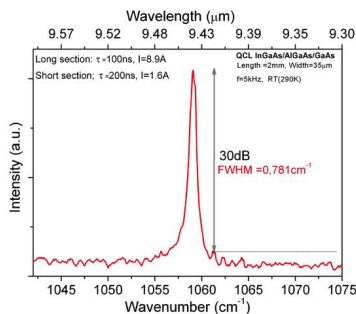


Fig.2. Room temperature single mode emission spectra of FIB processed TS CC-QCL showing side mode suppression ~30dB

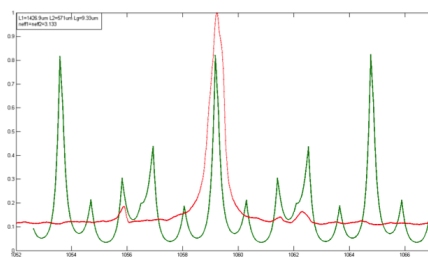


Fig. 3 Comparison of the experimental lasing spectrum (red) and calculated spectrum (green) of TS CC QCLs.

The observed mode position and mode spacing are in good agreement with the calculated values. The simple numerical model was applied to explain positions and mode spacing in the experimental data. The TSCC-QCL is treated as two coupled Fabry-Perot cavities. The transmission of each cavity was calculated taking into account its length and refractive index. The product of the resulting transmission curves together with the experimental lasing spectrum is plotted in Fig. 3. Positions and shapes of the peaks in simulated curve agree well with the experimental data.

This work was partially supported by PROFIT PBS 2/A3/15/2013 and by National Science Centre grant SONATA no. 2013/09/D/ST7/03966.

Reflectivity-based characterization of doped layers in the infrared device structures

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During the last years mid-infrared semiconductor lasers have continuously increased their application range to include gas sensing for detection and control of the presence and concentration of harmful gases like CO₂, SO_x and NH₃. The so-called GaSb-based interband cascade lasers (ICLs) are able to cover spectral range between 2 and 5 μm . In order to extend the region of interest to longer wavelengths indium arsenide substrates are required [1]. Furthermore, an InAs/AlSb superlattice cladding region, based on index-guiding method, well-liked in GaSb-ICLs, is no longer suitable due to stronger absorption in mid and far-infrared, thus it is replaced by a highly doped InAs layer. This plasmon-enhanced waveguide approach reduces cladding thickness that leads to enhanced heat dissipation and shorter growth time.

On the other hand, the doped InAs layers are utilized as sources of terahertz radiation that base on the photo-Dember effect. Such sources are demanded for THz imaging and gas sensing applications, just to name a few.

In order to precisely control and optimize the doping level in the InAs layers of both types of device structures, it is very helpful to have a measurement method of the carrier concentration. It has been already reported that by means of reflectance measurements the identification of a minimum in the spectrum near the plasma frequency, i.e. the so-called plasma-edge, allows for the estimation of carrier concentration through the Berreman effect [2]. Although this method provides a dielectric response in a quantitative manner, it is generally difficult to analyze the spectra if the structure is multi-layered. A novel approach to carrier concentration's determination is fast differential reflectivity (FDR) [3]. Due to its differential nature, this technique is sensitive to various optical transitions and other singularities in the dielectric function, and eliminates background signals related to the material features and set-up characteristic. The standard reflectance approach involves measurement of two orthogonal polarizations, p and s , in order to precisely locate the plasma-edge in vastly reach spectrum, whilst FDR technique provides direct information through resonance corresponding to a Berreman minimum. As a result, there is no polarizer requirement. Furthermore, FDR is a non-destructive technique with average measurement time as short as 1 min. FDR measurements were performed on a set of samples, including highly doped InAs layers and ICL structures, and based on that the related carrier concentrations could be determined in different kind of devices.

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[2] R. T. Hinkey et al., Journal of Applied Physics **110**, 043113 (2011)

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Calculations of infrared absorption in InAs/GaSb superlattices

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InAs/GaSb type-II superlattices offer enormous possibilities of application as the detector and laser structures with tailored wavelength in the infrared range [1-3]. The ability to absorb (or emit) infrared radiation by a superlattice (SL) is a key feature, which decides about the use of the structure for the optoelectronic devices. Within this work we performed calculations of the band structure and absorption coefficient of $(\text{InAs})_m/(\text{GaSb})_m$ SLs. The electron and holes states in the SL minibands CB_1 , HH_1 , LH_1 , CB_2 , HH_2 and LH_2 have been determined. We have used the four-band $\mathbf{k}\cdot\mathbf{p}$ method in which the nonparabolicity, strain and interface (IF) effects were taken into account [4-6]. Calculated electron and hole states allowed us to determine energies of possible optical transitions. Figure 1 shows the results of simulations obtained for the 10/10ML SL with asymmetric IFs. Calculations of the absorption coefficient were performed for the light of TE (i.e., Transverse Electric) polarization mode, incident perpendicularly to the SL layers. Results of calculations were compared to the absorption spectra presented in the literature [7, 8].

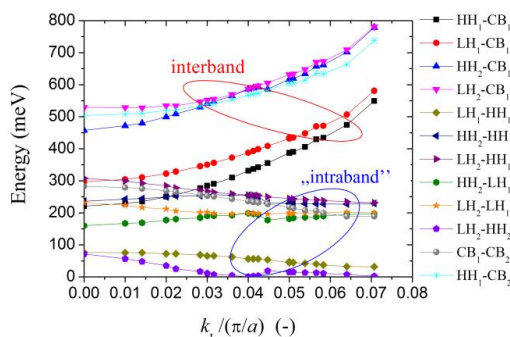


Fig. 1 Energies of possible optical transitions in $(\text{InAs})_{10\text{ML}}/(\text{GaSb})_{10\text{ML}}$ SL with asymmetric IFs. Calculations were performed with the use of the four-band $\mathbf{k}\cdot\mathbf{p}$ method. Presented relationships $E(k_{\parallel})$ describe the interband and „intraband” (i.e., between the valence band subbands and between the conduction band subbands) transitions in the SL at the temperature of 300K.

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Novel design of type-II “W” quantum wells for mid-IR emission with tensile –strained GaAsSb layer for confinement of hole

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Semiconductor diode lasers operating in the mid-infrared (MIR) are desirable for many applications in medical diagnostics, trace-gas analysis, pollution monitoring and molecular spectroscopy. This is due to strong absorption lines of many important industrial and environmental gases such as CO, CH₄, N₂O, NH₃ and others [1]. In this wavelength range there has been reported a significant progress in concepts of sources of coherent radiation in the MIR. Among them, one can distinguish mainly the laser diodes base on type I quantum wells (QWs), quantum cascade lasers (QCLs) and interband cascade lasers (ICLs). ICLs have several advantageous features, like broad tuning range of the emission [2], [3], minimized influence of the Auger related carrier losses [4], and a very low power consumption [5]. However, in order to fully exploit the potential of the ICLs, many parameters of these multilayer structures must be optimized, especially on the side of the active region which is composed of a cascade of type II QWs made of a broken gap materials.

In this work, we discuss a possibility of implementing type-II W-design QWs which allows preserving the large optical matrix elements in spite of indirect in the real space character of the optical transition. We have modelled the electronic structure properties of AlSb/InAs/GaAsSb/ InAs/AlSb QWs grown on GaSb substrate. The calculations have been carried out within the eight-band k·p theory including strain. As the GaAsSb layer is tensely strained this is possible to tune between the system with the heavy or light hole character of the fundamental optical transition. This provides an additional engineering parameter which eventually offers several potential benefits, also due to possible utilization of thicker QW layers in such a system: (i) enhanced the transitions oscillator strength, (ii) broad range of spectral tunability of the emission, (iii) less sensitive the transition energy or emission wavelength to the layer thickness inaccuracy. Therefore, using a type II structure with tensely-strained GaAsSb layers can be a promising way to improve the performance of the existing ICL devices or to extend their range of operation into longer wavelengths.

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7 March (Saturday)

Sessions

Sources, detectors, materials III

Applications IV

Fast response photodetectors for mid-infrared laser-based gas sensing

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We report on recent progress in fast response HOT (Higher Operating Temperature) photodetectors for mid-infrared laser-based gas sensing. The devices are typically based on complex HgCdTe heterostructures grown by MOCVD, MBE or LPE epitaxy. Artificial superlattice narrow gap semiconductors seem to be the most likely candidate to replace HgCdTe.

For a long time, photoconductive and photoelectromagnetic devices operating at near-room temperature have been used as fast (sub-nanosecond) photodetectors. Unfortunately, the devices suffer from modest detectivity. Recent efforts are focused on heterojunction photodiodes that potentially offer both fast response and high signal-to-noise.

This paper presents computer simulations and experimental studies that reveal mechanisms limiting time response. Design rules for unbiased or reverse biased photodetectors that are necessary to achieve fast response are described. The devices could be optimized for any wavelength within 2 to 16 μm range of infrared spectrum. In addition, optimization of associated electronics is also addressed.

The main mechanisms that determine time response of the devices is recombination of photogenerated charge carriers, their ambipolar drift and diffusion to the contacts and diffusion capacitance combined with built-in or external series resistance.

Practical photodetectors based on HgCdTe or 3-5V superlattice materials and detection modules for various types of laser-based gas sensing systems are described.

Keywords: HOT photodetectors, HgCdTe, mid-infrared, fast response detectors, laser gas sensing.

High-performance GaSb-based Interband Cascade Lasers

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A multitude of absorption lines of important gases is located in the infrared region between 3 μm and 6 μm , making it very interesting for tunable diode laser spectroscopy (TDLS). However, the realization of semiconductor lasers that operate cw at room temperature (RT) in this region has been quite challenging. Extending the emission of diode lasers beyond 3 μm is difficult due to the strong increase of Auger recombination and loss of carrier confinement. Quantum cascade lasers on the other hand perform very well above 4 μm , but the available conduction band offsets limit their emission on the short wavelength side. In recent years however, several breakthroughs have allowed the demonstration of good laser performance in the 3-6 μm region [1]. The most significant development in this regard is the interband cascade laser (ICL). This device combines features from diode and quantum cascade lasers and has shown good performance (including cw operation at RT) in the 3-5.5 μm range after major design optimizations [2]. In my talk, I'll discuss recent developments of GaSb-based ICLs. These include the demonstration of ICLs with very low threshold current densities [3], distributed feedback lasers with output powers larger than 20 mW and operation up to 80°C (see fig. 1a), single mode emission at 5.2 μm (see fig. 1b) for sensing applications [4] and distributed feedback lasers based on lateral metal gratings [5].

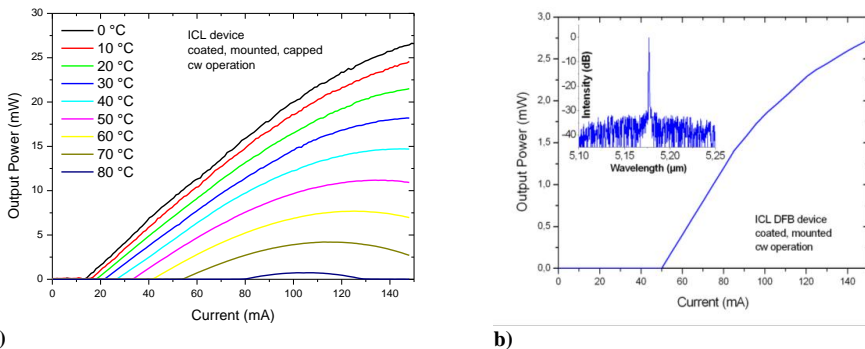


Fig. 1: a) Output power characteristic of a 3.5 μm ICL-DFB at different temperatures
b) Output power characteristic and emission spectrum of an ICL-DFB emitting at 5.2 μm

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Frontiers of QC Laser spectroscopy for high precision isotope ratio analysis of non-CO₂ greenhouse gases

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An important milestone for laser spectroscopy was achieved when isotope ratios of trace gases were reported at precision levels that allow addressing research questions in environmental sciences, inaccessible by the well-established isotope-ratio-mass-spectrometry (IRMS) method. For quantitative detection of isotopic signatures in ambient air, it is most often necessary to reach an isotope ratio precision of < 0.1 ‰. Although, this is well established for CO₂ isotopologues using quantum cascade laser absorption spectroscopy (QCLAS) [1,2], applications of QCLAS for N₂O and CH₄ stable isotopes are considerably more challenging, especially for the less abundant species, such as N₂¹⁸O and CH₃D.

Here, we review our recent developments on high precision isotope ratio analysis of non-CO₂ greenhouse gases, with special focus on the isotopic species of N₂O and CH₄. Furthermore, we show environmental applications illustrating the highly valuable information that isotope ratios of atmospheric trace gases can carry. For example, the intramolecular distribution of ¹⁵N in N₂O gives important information on the geochemical cycle of N₂O, while the analysis of the most abundant methane isotopologues ¹²CH₄, ¹³CH₄ and ¹²CH₃D are used to disentangle the various source/sink processes. We apply fast sweep direct absorption technique using single or dual QCL configuration, coupled into an astigmatic multipass cell with up to 204 m optical path length. For high precision (< 0.1 ‰) measurements in ambient air, it is necessary to enhance the mixing ratio of trace gases, especially for the rare isotopologues. This is achieved on-site by means of a recently developed fully automatic preconcentration unit [3], yielding a pre-concentration factor up to 500 and sequential desorption of CH₄ and N₂O.

Applications using the above instrumentation include the study of microbial processes in soil incubation experiments and waste water treatment [4]. The most recent study aimed at source partitioning between nitrification and denitrification pathways at a grassland field site based on high precision real-time analysis of $\delta^{15}\text{N}$ -N₂O. The intermolecular distribution of ¹⁵N substitutions (site preference, i.e. ¹⁴N¹⁵N¹⁶O versus ¹⁵N¹⁴N¹⁶O) and the oxygen isotopic composition ($\delta^{18}\text{O}$) of N₂O were measured with a precision of < 0.1 ‰. Furthermore, the laser based technique was validated with respect to IRMS, and has shown to be more precise and accurate, especially for the site-specific composition [5]. In a similar, dual QCL setup, we obtained a precision of 0.1 ‰ for both $\delta^{13}\text{C}$ and δD in 900 ppm CH₄ at 10 min spectral averaging. Currently, the new analytical system is being investigated in an extensive inter-comparison field campaign and compared to IRMS and cavity enhanced spectroscopic techniques.

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Hyper-Spectral-Imaging Applications on the Micro scale with Quantum Cascade Lasers

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Quantum Cascade Lasers (QCL) were used first as a tool for hyper-spectral-imaging in macroscopic applications such as stand-off explosive detection and detecting hazardous gas clouds. On the other end of the scale, scanning near field microscopy (SNOM/SNIM) has been used very successfully to investigate graphene and biological samples at nanometer resolution.

Using QCL illumination in a MIR-microscope set-up increases optical power leading to diffraction-limited spatial resolution, large fields of view, and drastically shorter acquisition times. The much broader fields of view compared to Scanning Near Field Optical Microscopes (SNOMs and SNIMs) are especially well-suited for many industrial and medical applications. The power levels are high enough to achieve fast response far above the noise level of detectors that don't require liquid nitrogen cooling. Because of the increased power density, the light can be spread across more pixels achieving the same signal level on the detector. This results in a very high spatial resolution leading to much more precise images of the sample. 480 x 480 pixels, each representing 1,4µm pixel size on the object are by far sufficient for a huge amount of applications. (see fig. 1).

Stitching algorithms can be still used to enlarge the image area. In this case the speed of acquisition is the decisive factor as the number of acquisitions rise with the square of the width of the area of investigation.. However, with discrete frequency illumination enabled by the tunable source, real time live images allow the investigation of dynamic events at video frame rates. Scanning the wavelength to appropriate values leads to a series of single frequency absorbance images that can be assembled into hyperspectral data cubes and chemical maps, all without the use of labels, stains, or tags. .

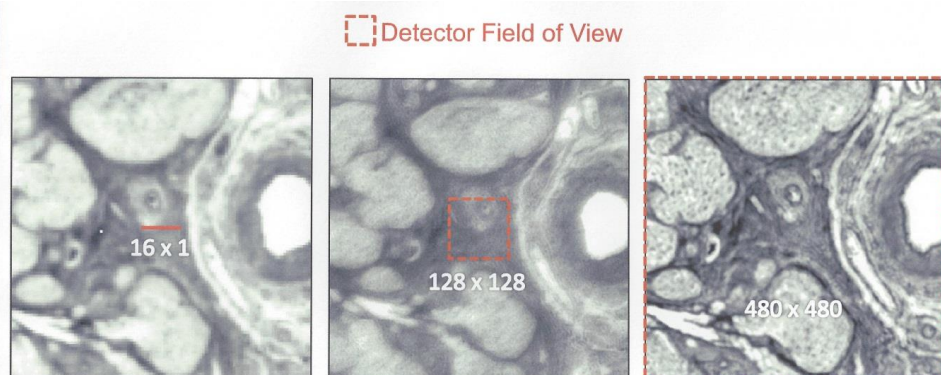


Fig. 1. High resolutions field of View: Linear array FTIR 6.3µm pixels linear Sweep (left)
Focal Plane Array FTIR 1.1 pixels 10x10 mosaic (middle)
Spero 1.4µm pixels, single frame (right)

(Tissue sample courtesy of Dr. Nora Laver, Tufts University)(Image courtesy of Dr. Michael Walsh, University of Illinois at Chicago)

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Open path gas sensing applications in Mid-IR using the GasEye tunable laser spectrometer

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Introduction. Recently Tunable Laser Spectroscopy (TLS) has extended its capabilities into the mid-IR region offering many new opportunities for industrial applications [1-3]. These applications require reliable unattended operation with service intervals of several years. Until now the lack of suitable laser sources in the main hydrocarbon absorption bands has limited the applicability of TLS in the chemical industry. Tunable mid infrared laser sources in the wavelength range 3.3 – 7.0 μm , utilizing novel Interband Cascade Laser (ICL) structures, are now available. This enables the development of novel photonic gas sensors for applications in process analytics and emission monitoring. The GasEye is a flexible gas analyzer platform developed by Airoptic. Utilizing the ICL as a laser source, open path sensor systems for the remote detection of formaldehyde and ethanol have been realized using the GasEye platform.



Monitoring of formaldehyde emission in wood board manufacturing. Annual worldwide production of formaldehyde is around 21 million tons. About half of this is used to make formaldehyde resins used as extremely strong and permanent adhesives in the majority of wood-based panels. Formaldehyde is also used in consumer products, such as shampoo and permanent press clothing. In homes, the most significant sources of formaldehyde are likely to be pressed wood products made using adhesives that contain formaldehyde. Current standards for formaldehyde in composites are 50 ppb issued by California Air Resources Board (CARB). Accurate on-line monitoring will allow manufacturers to gain tighter control of emission levels, reduce rejected lots, reduce claims, and improve profitability. Moreover, many workers in workplaces such as sawmills, plywood plants and furniture plants may be exposed to high levels of formaldehyde containing wood dust. Ultimately, real-time formaldehyde emissions monitoring will improve indoor air quality. Airoptic has developed the GasEye HCHO, a field instrument for formaldehyde monitoring, utilizing a 3599 nm ICL. The platform is self contained and includes signal processing electronics, laser driver, TEC driver and power supply. The optics is designed to facilitate the alignment of the beam onto a retro reflector while keeping the optical noise very low. The alignment is guided by a red aiming laser.

Stand-off monitoring of alcohol vapors in vehicles. There is a great need to increase the efficiency of the enforcement of the drunk-driving regulations. The randomness of the screening of potential offenders made at sobriety check-points is presently not satisfactory. A remote stand-off detection system for measuring the amount of ethanol vapor in the passenger compartment of a passing car has been developed around the GasEye platform. A laser beam at a wavelength around 3.3 μm is passed through the side window of a passing car. Airoptic has developed a signal processing schemes allowing to perform a normalized measurement with good selectivity. The system has been verified on vehicles in an outdoor setting.

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Isotope Metrology Using Mid-IR Spectroscopy

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The ability to determine the isotopic composition of a gas sample can provide valuable information about the emission source of that gas species. Such information is relevant not only for atmospheric studies, but for industrial applications as well. Isotopic information has traditionally been determined by mass spectrometry, which is a well-established technique capable of high precision measurements. However, with long sample preparation and analysis times for some species, and the complexity of the instrumentation, its use is limited to the laboratory. This places restrictions on the achievable data acquisition rates and the potential applications.

With the desire for real-time, field capable isotope analysis methods, optical detection techniques are increasingly being employed. The Centre for Metrology and Accreditation (MIKES) is utilising optical-detection based techniques such as cavity ring-down spectroscopy to develop such instrumentation. Recent advances in Mid-IR laser technology have produced commercially available compact and easy to use laser sources, allowing for spectrometers with a compact setup which can be used for fast, on-site measurements.

Our current research has focused on carbon dioxide (CO₂) and methane (CH₄). We are developing a carbon isotope spectrometer to detect radiocarbon (¹⁴C), permitting the determination of the ¹⁴C/¹²C ratio of a sample. The spectrometer has successfully monitored elevated levels of ¹⁴C, with the instrument sensitivity now being enhanced to detect concentrations of ¹⁴C below natural abundance levels. This will allow for its use in applications such as the determination of the biological origin of fuel, carbon capture and storage leaks detection or drug development using radiocarbon labelled drugs. In addition, a spectrometer capable of measuring the ¹³C/¹²C and D/H isotope ratios of CH₄ in ambient air in real time is under development.

MIKES has strong connections with other European National Measurement Institutes, and is contributing to reference gas and transfer standard metrology through the development of instruments for stable isotope measurements.

Laser spectroscopic CO measurements in the near and mid infrared regions

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Carbon monoxide is an atmospheric molecule that has an indirect effect on global warming, and an important molecule for industrial process control and environmental monitoring. Laser spectroscopic techniques such as tunable diode laser absorption spectroscopy (TDLAS) and quantum cascade laser absorption spectroscopy (QCLAS) have been shown to be capable of accurate measurements of CO amount of substance fractions in various gas analysis applications [1][2].

In this work, we present our approach for absolute laser spectroscopic CO amount of substance fraction measurements in view of atmospheric applications, using near and mid infrared lasers. We elaborate on the current limitations in our CO amount fraction measurement results to meet, e.g., the compatibility goal of 2 nmol/mol stated by the World Meteorological Organization (WMO) for atmospheric CO measurements [3], and outline possible solutions. In addition, a discussion on achieving direct traceability of laser spectroscopic CO amount of substance fraction results to the international system of units (SI) and the estimation of uncertainties compliant with the Guide to the Expression of Uncertainty in Measurement (GUM) [4] is presented.

Acknowledgement

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Laser calorimetry spectroscopy for in-liquid dissolved gas detection and measurement

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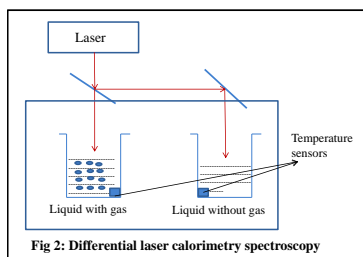
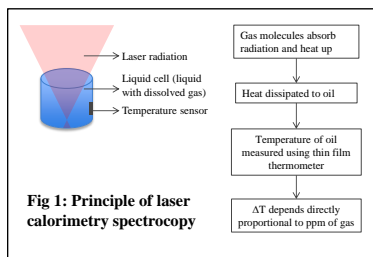
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Analysis of dissolved gases in liquids is of prime importance in a wide array of fields like transformer health monitoring, food and beverage industries (e.g. measuring amount of ethanol in wine), oil and gas industry (measuring levels of moisture in natural gas, etc.). In many of these fields chemical and electrical analysis cannot be used (electrical is ruled out due to harsh environmental conditions and due to safety requirements, while in many cases chemical does not have enough specificity). In most cases, gas chromatography (GC) - both offline and online has been used as the best possible solution. Some forms of optical analysis is preferred due to its specificity and its non-interfering nature. However, optical spectroscopies suffer from need for line of sight, and if measured in liquid, the liquid should be transparent or substantially transparent to the radiation (wavelengths) used. For liquids like water or oil, finding a wavelength where the gas absorbs strongly while the liquids do not absorb is very difficult. Usually the absorbance of the liquid is much stronger than absorbance of the gas. Therefore conventionally, gases are extracted out from the liquids and then analyzed, either using an optical method or using GC. There is, therefore, an overwhelming need for in-liquid dissolved gas analysis.

Here we present a novel technique - laser calorimetry spectroscopy (LCS) [1,2], which is a combination of laser absorption spectroscopy and calorimetry, for the in-liquid detection of gases dissolved in liquids. The technique involves determination of concentration of a dissolved gas by irradiating the liquid with light of a wavelength where the gas to be detected absorbs, and measuring the temperature (T) change caused by gas absorbance (fig 1). The T change due to the absorbance of the liquid is eliminated (or substantially eliminated) by using a differential T measurement (using a reference liquid) (fig 2). Since most gases that need to be detected have their fundamental vibrational modes in the MIR region, QCLs and ICLs have been used for the measurements. Using LCS, we have been able to detect ppm levels of gases (like C₂H₂ and CO₂) without extracting them from the liquid. The fundamentals of LCS technique, experimental details, proof of concept experiments and some results will be presented.



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Selective and Sensitive VOC Breath Analysis Using a 3.3 μm Broadly-Tunable VECSEL

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To date, more than 3000 different volatile organic compounds (VOCs) have been identified in human breath, some of which serve as important biomarkers for different physiological processes and diseases. In this work, we address the spectroscopic detection of such compounds using a novel broadly tunable mid-infrared Vertical External Cavity Surface Emitting Laser (VECSEL) [1]. It can be scanned at high rate (1 kHz) over more than 50 cm^{-1} and has an emission bandwidth centered at $3.3\text{ }\mu\text{m}$ (3000 cm^{-1}), which well coincides with the fundamental stretching vibrational band of the C-H bond. Thus, it can probe the absorption features of hydrocarbons, aldehydes, alcohols, ketones and many other organic molecules. We demonstrate the suitability of this laser source for the measurement of acetone, which is a by-product of ketogenesis and a possible biomarker for Type 1 diabetes and dietary energy-balance.

For the sake of simplicity and cost efficiency, we opt for purely spectroscopic detection without any sample preparation or water vapor removal. Despite largely dominating contribution from water and methane, our system is able to reliably and accurately detect 2 ppm acetone in a breath-like mixture of 3.8% water vapor and 1.8 ppm methane, as demonstrated in Figure 1. The achieved 1σ detection limit for acetone is 170 ppb after 5 s, and it reaches about 25 ppb after 5 minutes of averaging.

These promising results imply a further development of the instrument towards a demonstrator that will be used in a clinical study for prevention of obesity through individual energy balance monitoring.

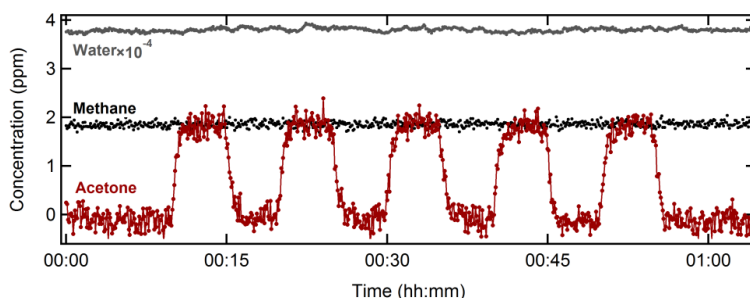


Figure 1. Experimentally measured sequential variation of acetone concentration between 0 and 2 ppm (red) in a breath-like matrix containing 3.8% water (grey) and 1.8 ppm methane (black). The gas mixture was prepared using calibration gases and humidified in water bath at $40\text{ }^{\circ}\text{C}$.

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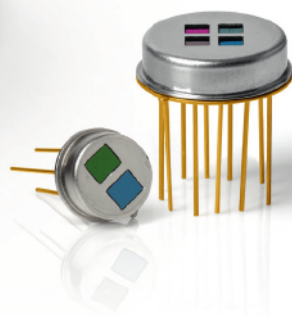
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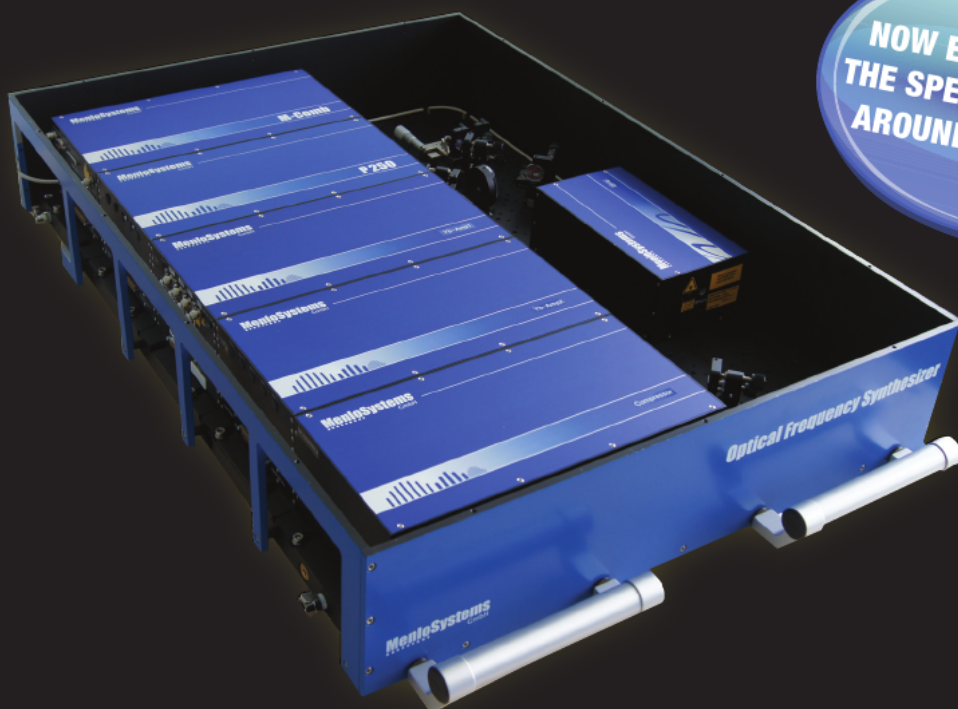
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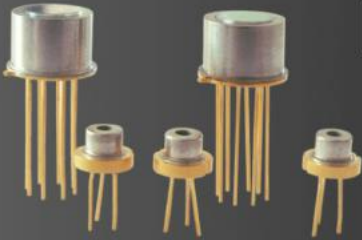
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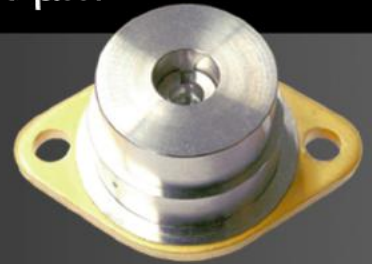
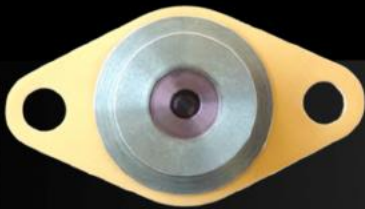
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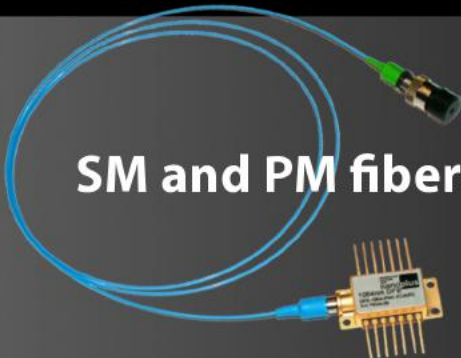
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