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

# mirsens2

OCTOBER 18-20 2012
Institute of Physics
Wrocław University of Technology

# Book of Abstracts

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**International Workshop on Opportunities and Challenges in Mid-Infrared Laser-based Gas Sensing**

# mirsens2

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| Workshop Venue: WRUT Conference Center (building D-20), Z. Janiszewskiego 8 str. | | | |
|---|---|---|---|
| 8.50 | 9.00 | Opening Address | |
| *SESSION:* **Lasers 1** | | | |
| 9.00 | 9.45 | **J Faist** | Mid-infrared quantum cascade lasers combs for spectroscopy |
| 9.45 | 10.30 | **C Grasse** | GaSb- and InP-based devices for mid-infrared gas sensing application |
| 10.30 | 10.45 | Coffee break | |
| *SESSION:* **Industrial advertisements** | | | |
| 10:45 | 11.00 | **A Vizbaras** | New player in the mid-IR market: Company overview |
| 11.00 | 11.15 | **D Stanaszek** | Uncooled IR detectors for gas sensing |
| 11.15 | 11.30 | **I Z Kozma** | Mid-IR frequency combs |
| 11.30 | 11.45 | **P M Moselund** | Broadband and tunable light sources from the visible to the IR |
| 11.45 | 12.00 | **J Koeth** | Introducing novel DFB devices for sensing in the MIR between 3.0 and 3.5 µm |
| 12.00 | 13.00 | Lunch | |
| *SESSION:* **Lasers 2** | | | |
| 13.00 | 13.20 | **P M Moselund** | MIR supercontinuum a new high intensity source for MIR spectroscopy |
| 13.20 | 13.40 | **N Hattasan** | InGaAsSb/GaSb lasers and photodetectors integrated on a SOI waveguide circuit for spectroscopic applications |
| 13.40 | 14.00 | **J Wueppen** | Ultrafast mid-IR laser source with a tuning range from 9 to 16 microns based on nonlinear frequency conversion |
| 14.00 | 14.20 | **K Krzempek** | Mid-infrared optical frequency-comb synthesis using nonlinear optical conversion in PPLN crystals |
| 14.20 | 14.35 | Coffee break | |
| *SESSION:* **Applications 1** | | | |
| 14.35 | 15.20 | **H-J Wagner** | Broadband-tunable external-cavity QCL for the spectroscopic detection of hazardous substances |
| 15.20 | 16.05 | **P Kluczyński** | New gas sensing applications in Mid-IR |

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|---|---|---|---|
| 16.05 | 16.20 | Coffee break | |
| 16.20 | 16.40 | **E Pliński** | From MIR to FIR – physical chemistry vs chemical physics. Terahertz waves in sensing technology |
| 16.40 | 17.00 | **A Popescu** | Miniaturized TDLS optical gas sensing devices |
| 17.00 | 17.20 | **S Rhein** | Resolving methane isotopologues with a 3.3 μm DFB-diode laser using PAS |
| 17.20 | 17.40 | **M Nikodem** | Differential optical dispersion spectroscopy for high accuracy trace-gas sensing |
| 18.00 | 20.00 | Conference Dinner | |

| Workshop Venue: WRUT Conference Center (building D-20), Z. Janiszewskiego 8 str. | | | |
|---|---|---|---|
| *SESSION:* **Lasers 3** | | | |
| 9.00 | 9.45 | **M Razeghi** | Recent progress in mid-infrared laser sources at Center for Quantum Devices, Northwestern University |
| 9.45 | 10.30 | **J Abell** | Single-mode interband cascade lasers for chemical sensing |
| 10.30 | 10.50 | Coffee break | |
| 10.50 | 11.10 | **R Weih** | Interband cascade lasers with low threshold current densities |
| 11.10 | 11.30 | **H Moser** | Time-resolved spectral behavior of ring cavity surface emitting QCLs (RCSE-QCLs) |
| 11.30 | 11.50 | **K Kosiel** | High power GaAs/AlGaAs quantum cascade lasers |
| 11.50 | 12.10 | **M Fischer** | Specialized GaSb based laser sources for sensing: Devices offering an extended tuning range and devices with high optical output power |
| 12.10 | 13.10 | Lunch | |
| *SESSION:* **Materials** | | | |
| 13.10 | 13.55 | **G Sęk** | GaSb-based structures for mid-infrared emitting lasers: optical study |
| 13.55 | 14.15 | **Q Zhuang** | Dilute nitride InAsN multi-quantum wells on metamorphic buffer layer on InP for mid-infrared optoelectronics |
| 14.15 | 14.35 | **M Motyka** | Developments in Fourier-transform-based optical characterization of mid to far infrared semiconductor materials |
| 14.35 | 14.55 | **F Janiak** | Optical properties of InGaAsSb/InAs solid solution heterostructures grown by LPE |
| 14.55 | 15.10 | Coffee break | |
| *SESSION:* **Lasers 4** | | | |
| 15.10 | 15.55 | **R Q Yang** | InAs-based interband cascade lasers |

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|---|---|---|---|
| 15.55 | 16.40 | **D Sanchez** | Single mode cw operation of monolithic GaSb VCSELs |
| 16.40 | 17.00 | **P Fuchs** | Single mode quantum cascade lasers with shallow-etched distributed Bragg reflector |
| 17.00 | 17.20 | **M Dallner** | InAs-based interband-cascade-lasers for emission around 6 μm |
| 17.20 | 17.40 | **Ł Sójka** | Mid infrared fiber lasers |

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| **Workshop Venue: WRUT Conference Center (building D-20), Z. Janiszewskiego 8 str.** | | | |
| *SESSION:* **Applications 2** | | | |
| 9.00 | 9.45 | **F Tittel** | Mid-infrared semiconductor laser based trace gas sensor technologies: Recent advances and applications |
| 9.45 | 10.30 | **P Kaspersen** | Potentials of mid-infrared spectroscopy in emission control |
| 10.30 | 10.45 | Coffee break | |
| 10.45 | 11.05 | **P Fietzek** | Applying optical absorption techniques in the deep sea - underwater greenhouse gas measurements |
| 11.05 | 11.25 | **L-E Nilsson** | The use of MIR spectroscopy for localization of threat substances in urban societies |
| 11.25 | 11.45 | **A Miller** | Selective trace gas detection through CEAS and diffusion time of flight |
| 11.45 | 12.05 | **J P Waclawek** | QEPAS based sensor for detection of sulfur dioxide using a CW-DFB-QCL |
| 12.05 | 12.25 | **B Jankiewicz** | Measuring system for hazardous substances detection |
| 12.25 | 12.45 | **L Cocola** | A new method for absorption feature resolution from wavelength modulation spectroscopy (WMS) signals |
| 12.45 | 13.00 | Closing Address | |
| 13.00 | 14.00 | Lunch | |

Invited

# Mid-infrared quantum cascade lasers combs for spectroscopy

**J. Faist[1], A.Hugi[1], S. Riedi[1], A. Bismuto[1], B. Hinkov[1], E. Gini[2], S. Blaser[3] and M. Beck[1]**

[1] *Institute for Quantum Electronics, ETH Zürich, 8093 Zürich, Switzerland*
[2] *FIRST Center for Micro- and Nanoscience, ETH Zürich, 8093 Zürich, Switzerland*
[3] *Alpes Lasers, Rue Champreyvres, Neuchatel, Switerland*

The quantum cascade laser (QCL)[1] has demonstrated operation over an extremely wide wavelength range extending from the mid-infrared at 2.9 m to the Terahertz at 360 m. In addition, quantum cascade lasers operating in the mid-infrared have now demonstrated extremely high powers (>1W c.w.) with increasingly high wallplug efficiencies (>25%)[2, 3]. As shown in Fig. 1, in a new development, ultra low dissipation devices have been fabricated, operating with powers below 1W at room temperature.

The QCLS have attracted great scientific and industrial interest in covering the mid-infrared or fingerprint region of the electromagnetic spectrum with these broadly and quickly tunable light sources. Such sources would greatly facilitate the optical chemical sensing of mixture of gases or liquids.

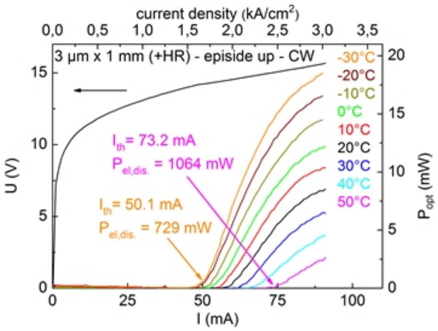



**Fig. 1** Light-current-voltage characteristics of a typical episide up mounted device (3 μm x 1 mm +HR-backside coating) in CW-operation at different temperatures.

In particular, there is continuous interest in using External Cavity-Quantum Cascade Lasers (EC-QCLs) for trace gas analysis [4] because EC-QCLs offer a broad tuning range and are continuously tunable. The tuning speed is in principle only limited by the achievable grating rotation of the EC-QCL. Another advantage is the possibility to tailor the wavelength of the gain chip using quantum engineering. We have demonstrated recently the broadband tuning of a EC-QCL with a five-stack active region over 440cm$^{-1}$[5].

In one extension, we have redesigned the broadband active region to operate with only three stacks. The lower applied bias allows a similar tuning range (445cm$^{-1}$, as shown in Fig. 1a) but enables continuous wave operation. As shown in Fig. 2b), a newly developed 3 m active region was also tested in pulsed mode and achieved a 243cm$^{-1}$ tuning range.

As shown in Fig. 3, the interesting feature of the new three stack active region is its capability to operate in continuous wave and to provide a very wide band spectrum extending over more than 250cm$^{-1}$. In particular, the modes are extremely narrow (below the

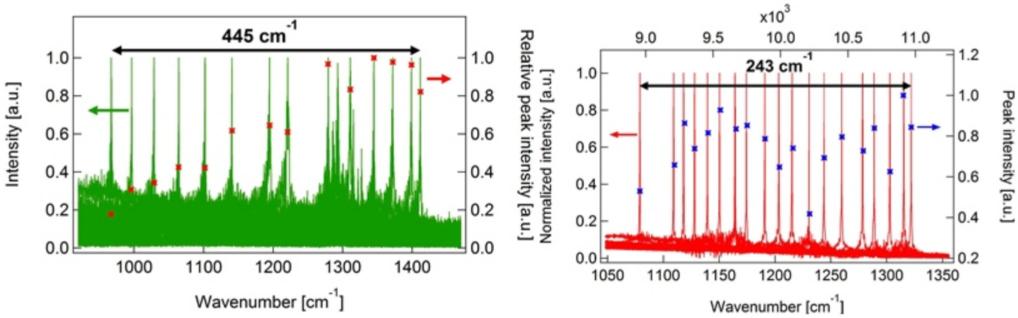



**Fig. 2** a): Tuning of a buried heterostructure EC-QCL using a gain element with a three stage heterogeneous cascade. Right: Pulse mode tuning of a 3um device.

resolution limit of the high resolution FTIR), as show in Fig. 4. In a new development, we will discuss the coherent properties of these modes using spectroscopy techniques [6].

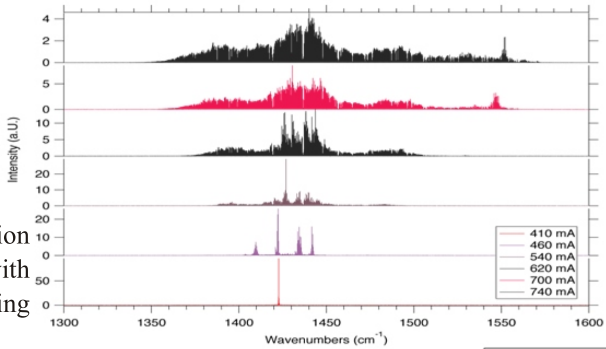
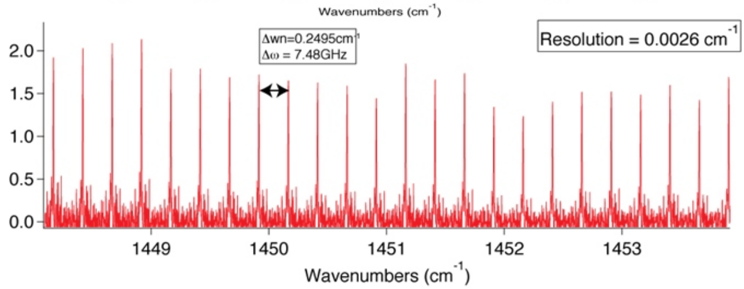



**Fig. 3** a) Continuous wave operation of a broadband device, with multimode operation extending over more than 250cm$^{-1}$.




[1] J. Faist, F. Capasso, D. Sivco, C. Sirtori, A. Hutchinson, and A. Cho, *Science* **264**, pp. 553-556 (1994).
[2] R. Maulini, A. Lyakh, and A. Tsekoun, *Optics Express* **19**, 17203 (2011).
[3] Y. Bai, N. Bandyopadhyay, S. Tsao, S. Slivken, and M. Razeghi, *Appl. Phys. Lett.* **98**, 181102 (2011).
[4] A. Kosterev, G. Wysocki, Y. Bakhirkin, S. So, R. Lewicki, M. Fraser, F. Tittel, and R.F. Curl, *Appl Phys B-Lasers O* **90**, pp. 165-176 (2008).
[5] A. Hugi, R. Terazzi, Y. Bonetti, A. Wittmann, M. Fischer, M. Beck, J. Faist, and E. Gini, *Appl. Phys. Lett.* **95**, 061103 (2009).
[6] A. Hugi, G. Villares, S. Blaser, H.C. Liu, J. Faist, accepted *Nature*.

Invited

# GaSb- and InP-based devices for mid-infrared gas sensing application

**C. Grasse, S. Sprengel, K. Vizbaras, M.-C. Amann**

*Walter Schottky Institut, Technische Universitaet Muenchen, Am Coulombwall 4, 85748 Garching, Germany*

In recent years, applications like gas sensing and environmental monitoring have considerably raised the demand on mid-infrared laser devices. For environmental critical gases like $CO_2$ or $CH_4$ it is especially important to cover the wavelength range between 2 and 4 µm due to their strong absorption lines in this region [1].

Since the emission from type-I quantum wells of the mature InP-based material systems is limited to around 2.3 µm due to strain restrictions, GaSb-based devices are usually deployed to address wavelengths over 2.3 µm. However, exceeding the emission beyond 3 µm is also for this material system very challenging due to increasing Auger recombination, free carrier absorption and decreasing hole confinement [2]. Hence, in the past alternative device concepts like interband cascade lasers (ICL) with type-II active regions were the only choice to exceed the emission, but with the drawback of increased voltage drop due to the cascading [3]. But improve-ments of the growth technique and the introduction of quinternary AlGaInAsSb-based barriers have recently

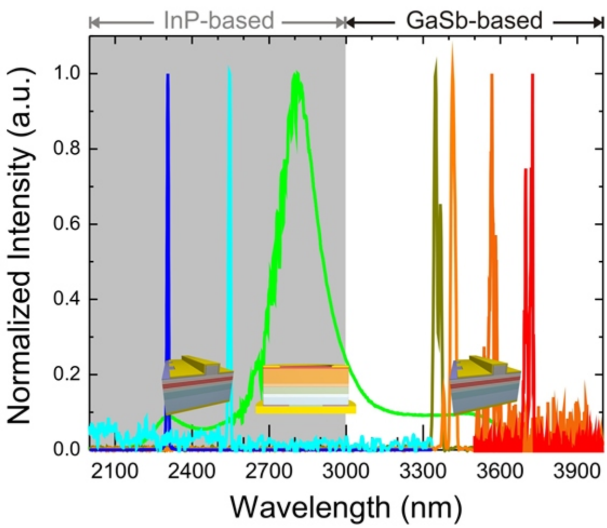



**Fig. 1** Emission spectra of InP-based edge emitters and a resonant cavity LED are shown. Beyond 3µm, spectra of GaSb-based edge emitters are illustrated (all edge emitters operate in pulsed mode at room-temperature).

yielded up to 3.7 µm emission wavelength at room-temperature with standard type-I edge emitters (see Fig. 1), which are offering low power consump-tion and a theoretically possible emission up to 4 µm [2].

Furthermore we present the first electrically pumped interband emission up to 3 µm with InP-based type-II GaInAs/GaAsSb heterostructures as shown in Fig. 1. Similar to ICLs the spatial separation of holes and electrons is used here to overcome the bandgap limitations of the material. This offers the possibility to implement the well-established and mature InP-based device technology into the desired wavelength range and gain from the superior physical properties like the high thermal conductivity. Laser performance has already demonstrated up to 2.5 µm and given the fact that the presented structures are still at an early stage [4], therefore, the recent developments are promising to enter further into the mid-infrared wavelength range.

[1] L. Rothman et al., *J. Quant. Spectrosc. Radiat. Transf.* **110**, 533-572 (2009).
[2] K. Vizbaras et al., *Proceedings of the SPIE, vol.* **8277**, pp. 82771B-82771B-7 (2012).
[3] S. Höfling et al., *Proceedings of SPIE* vol. **8432**, 84320N (2012).
[4] S. Sprengel et al., *Appl. Phys. Lett.* **100**, 041109 (2012).

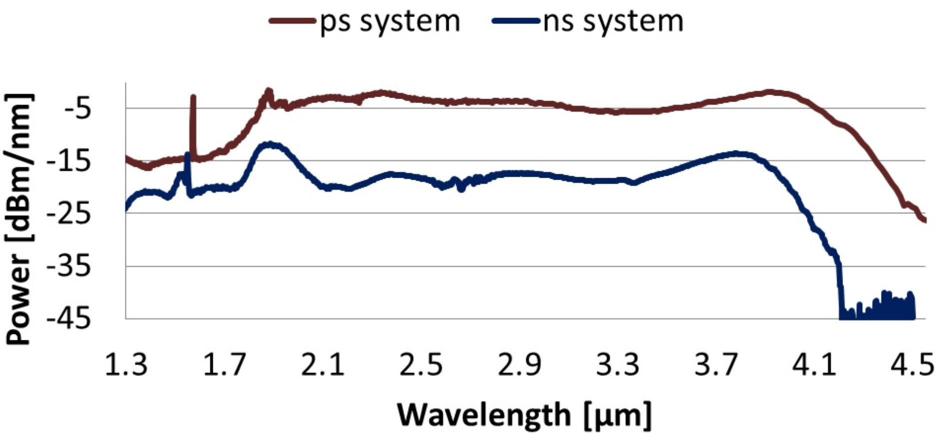
# MIR supercontinuum a new high intensity source for MIR spectroscopy

**Peter M. Moselund[1], Christian Petersen[1], Lasse Leick[1], Carsten L. Thomsen[1], Sune Dupont[2] and Soren R. Keiding[2]**

[1]*NKT Photonics A/S, Blokken 84, Birkeroed, Denmark*
[2]*Dept. of Physics and Astronomy, Aarhus Univ. Ny Munkegade 120, Aarhus C, Denmark*

We present the development of supercontinuum light sources for the mir-IR and as an example we show the mid-IR light sources developed at NKT as well as their application to mid-IR spectroscopy.

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. Based on the experience gained developing our market leading visible spectrum supercontinuum sources NKT Photonics has built mid-infrared supercontinuum sources based on both modelocked picosecond fiber lasers and ns lasers. The sources are pumped by a ~2 m laser based on a combination and ZBLAN fibers and generates up to 1 W covering the full spectrum of 1.5 – 4.5 µm spectrum in a single mode fiber format. We will present results obtained by applying the source for mid-infrared microscopy where absorption spectra can be used to identify the chemical nature of different parts of a sample and discuss the possible application of a mid-IR supercontinuum source in other areas. We will also briefly introduce MINERVA which is a EU FP7 program on mid-IR cancer diagnostics using MIR supercontinuum, starting in November 2012.



# InGaAsSb/GaSb lasers and photodetectors integrated on a silicon-on-insulator waveguide circuit for spectroscopic applications

**N. Hattasan[1], A. Gassenq[1], L. Cerutti[2], J.B. Rodriguez[2], E. Tournié[2], G. Roelkens[1]**

*[1]Photonics Research Group-UGent/imec, Sint-Pietersnieuwstraat 41, 9000 Gent, Belgium*
*[2]Université Montpellier 2 – CNRS, UMR 5214, Place Bataillon, 34095 Montpellier, France*
*Email: nannicha.hattasan@intec.ugent.be*

Spectroscopy is one of the most efficient techniques for the analysis of solid, liquid or gaseous samples, especially in the shortwave infrared region where water absorption is low. While conventional, bulky systems with expensive sources and detectors offer high-accuracy measurement results, compact and low-power systems are very attractive for many applications including portable gas sensors and implantable medical devices.

Silicon-On-Insulator (SOI) provides a platform for the realization of compact optical waveguide circuits with mass production capability by exploiting existing CMOS fabrication technologies. This results in low-cost integrated optical circuits. On the other hand, GaSb excels as an active component platform in the shortwave infrared wavelength range. It provides very efficient and room temperature operation lasers and detectors, making it ideal for spectroscopy [1]. Combining SOI and GaSb opto-electronic components would thus yield a compact, efficient spectroscopic detection system.

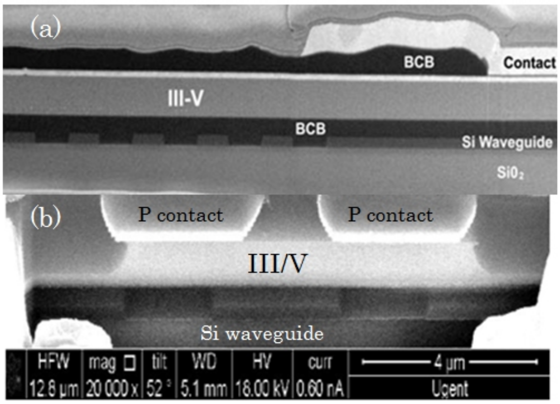



**Fig. 1** SEM image of the realized components a) grating-assisted coupling, b) evanescent coupling

In this paper, we report integrated GaSb-based photode- tectors on SOI and integrated Fabry-Perot (FP) lasers. The epitaxial layers are transferred onto the Si waveguide circuit by a die-to-wafer bonding process using Benzocyclobutene (BCB) as the bonding agent. 2 coupling mechanisms from the SOI waveguide circuit to the integrated photodetector are studied: evanescent coupling and grating-assisted coupling. High responsivity (>1A/W) of the evanescent photodiode and 0.4 A/W for grating-assisted coupling at 2.2µm is achieved. The dark current is ~4µA at -1V. SEM image of both designs are shown in Fig.1.

We also demonstrate thin-film GaSb-based FP lasers integrated on a carrier substrate. The device operates at 2.02 µm wavelength in continuous wave at room temperature. A threshold current of ~49.7 mA is achieved. This yields a current density of ~828A/cm$^2$. The integrated laser operates up to 35 $^o$C. Figure 2 represent the LIV characteristic of the laser and its spectrum is shown in the inset.

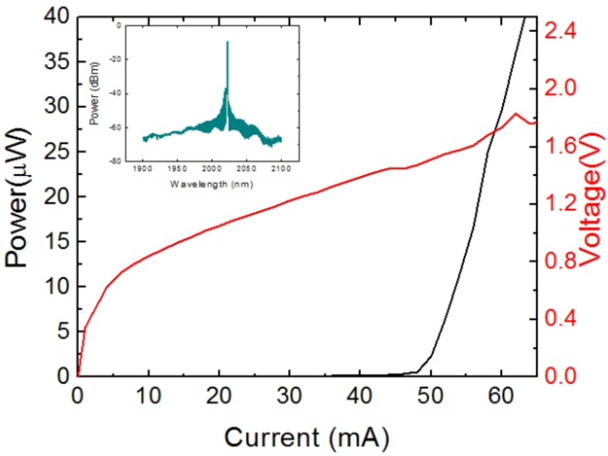



**Fig. 2** LIV spectrum of thr realised laser

[1] D. Barat, J. Angellier, A. Vicet, Y. Rouillard, L. Le Grateit, S. Guilet, A. Martinez, A. Ramdane, *Appl. Phys. B* **90**, 201 (2008).

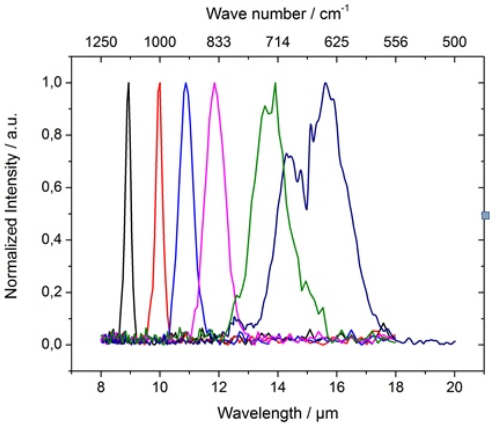
# Ultrafast mid-IR laser source with a tuning range from 9 to 16 microns based on nonlinear frequency conversion

**J. Wueppen[1], S. Nyga[1], T. Taubner[1, 2] and B. Jungbluth[1]**

*[1]Fraunhofer Institute for Laser Technology ILT, Steinbachstr. 15, 52074 Aachen, Germany*
*[2]RWTH Aachen University, I. Institute of Physics, Aachen, Germany*

Nonlinear optics is well known for the capability of generating output at every wavelength range from the deep UV to the far IR. The Laser system we propose (originally developed for spectroscopy of solids), could be interesting for gas sensing in the wavelengths ranges, where QCLs are not available commercially.

The Laser system consists of a commercially available ps-Laser as the pump source and two subsequent nonlinear converter steps. Laser and process parameters were chosen to allow a simple optical setup, which is easy to align and delivers a temporally stable output signal. The pump- Laser is a commercially available Nd:YVO4 mode-locked MOPA, providing 10 ps pulses at 1064 nm wavelength and a repetition rate of 20 MHz. The maximum average output power is 20 W. In the first conversion step, an optical-parametric generator (OPG), two waves are generated, tunable between 1.6 µm and 3 µm (signal wave < 2.28 µm < idler wave). These two waves are mixed in a difference frequency generator to generate the mid-IR waves, in these experiments tunable between 9 µm and 16 µm. The maximum output power measured so far was 10 mW.



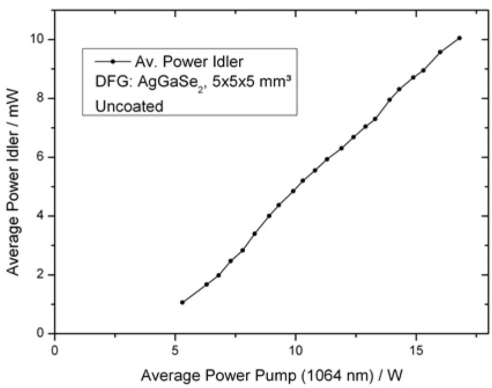




**Fig. 1** Generated spectrum from the second NLO converter (left side) and output power at these wavelengths (right side).

The wavelength range can be adapted to shorter or longer values by applying crystals with suitable phase-matching angles and adequate mirrors. The output is stable for hours and the beam pointing does not change at wavelength tuning. In a certain range the wavelengths can be tuned in milliseconds.

[1] A. Gambetta, R. Ramponi, M. Marangoni, "Mid-infrared optical combs from a compact amplified Er-doped fiber oscillator", *Optics Letters* **33**, No. 22 (2008).

[2] F. Rotermund, V. Petrov , F. Noack, "Difference-frequency generation of intense femtosecond pulses in the mid-IR (4–12 μm) using $HgGa_2S_4$ and $AgGaS_2$", *Optics Communications* **185**, 177 (2000).

# Mid-infrared optical frequency-comb synthesis using nonlinear optical conversion in PPLN crystals

**Karol Krzempek, Grzegorz Soboń, Krzysztof M. Abramski**

*Laser & Fiber Electronics Group, Wroclaw University of Technology, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Poland*

This talk will focus on our recent research devoted to generating mid-infrared (mid-IR) laser radiation with the use of difference frequency generation (DFG) effect occurring in periodically poled lithium niobate (PPLN) crystals. Novel, compact, single-active-fibre laser source for DFG processes will be presented [1].

Mode-locked fibre lasers are a hot topic for researchers all over the world. They have attracted even more attention after John L. Hall and Theodor W. Hänsch received a Nobel Prize in 2005 for their contribution to the development of the optical frequency comb technique. Both ultra-short pulses and characteristic comb-like optical spectrum, generated by mode-locked lasers, have found numerous applications in modern medicine [2,3], industry [3,4], metrology [5] and spectroscopy [6,7]. Targeting different applications requires a suitable wavelength of the mode-locked laser. Nowadays, we can observe an ongoing race to cover the whole optical spectrum with well-defined frequency combs. Fibre-based optical frequency combs working in the 1 μm and 1.55 μm wavelength region have proven to be reliable, robust and relatively low-cost sources of ultra-short pulses in this optical region.

In this presentation preliminary results of synthesizing an 3.39 μm mid-IR optical frequency comb using compact fibre based laser sources will be presented. In order to transfer the optical spectrum and time related characteristics of a mode-locked 1.55 μm center wavelength fibre laser a nonlinear DFG process occurring in PPLN crystals was used. The second source required for the DFG process was a single-mode 1.06 μm seed diode. Along with the "standard" DFG approach, utilizing two separate pumping lasers, a novel, integrated, single-active-fibre laser source generating both 1.55 μm and 1.06 μm radiation will be presented. The all-fibre dual-wavelength source uses a double-clad (DC) erbium-ytterbium co-doped fibre as the amplifying medium [1]. The residual 1 μm amplified spontaneous emission (ASE), normally present in the output spectrum due to the ytterbium doping, is used as the second pumping source in the DFG process.

[1] G. Sobon, P. Kaczmarek, K.M. Abramski „Erbium-Ytterbium Co-doped Fiber Amplifier Operating at 1550 nm with Stimulated Lasing at 1064 nm", *Optics Communications* Vol. **285**, Issue 7, pp. 1930-1934 (2012).

[2] M. J. Thorpe, D. Balslev-Clausen, M. S. Kirchner, and J. Ye, "Cavity-enhanced optical frequency comb spectroscopy: application to human breath analysis", *Optics Express* Vol. **16**, No. 4, 2387 (2008).

[3] F.H. Loesel et al., "Non-thermal ablation of neural tissue with femtosecond laser pulses", *Appl. Phys*. **B 66**, 121 (1998).
[4] I. Coddington, W. C. Swann, L. Nenadovic and N. R. Newbury, “Rapid and precise absolute distance measurements at long range”, *Nature Photonics* **3**, (2009).
[5] J. Ye, H. Schnatz, and L. W. Hollberg, “Optical Frequency Combs: From Frequency Metrology to Optical Phase Control”, IEEE Journal of selected topics in quantum electronics, vol. **9**, no. 4 (2003).
[6] A. Foltynowicz, P. Mas1owski, T. Ban, F. Adler, K. C. Cossel, T. C. Briles and J. Ye, “Optical frequency comb spectroscopy”, *Faraday Discuss.* **150**, 23–31 (2011).
[7] M. C. Stowe, M. J. Thorpe, A. Pe'er, J. Ye, J. E. Stalnaker, V. Gerginov, and S. A. Diddams, “Direct frequency comb spectroscopy”, *Advances in Atomic, Molecular and Optical Physics* **55** (2008).

Invited

# Broadband-tunable external-cavity quantum cascade lasers for the spectroscopic detection of hazardous substances

**J. Wagner, S. Hugger, F. Fuchs, Q. K. Yang, J.-P. Jarvis, R. Driad and R. Aidam**

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Broadband tunable external cavity quantum cascade lasers (EC-QCL) have emerged as attractive light sources for mid-infrared (MIR) “finger print” molecular spectroscopy for e.g. detection and identification of chemical compounds. Compared to Fourier Transform Infrared (FTIR) spectrometers EC-QCL offer the advantage of a much higher spectral brightness, i.e. high output power within a narrow wavelength interval, as well as a collimated low-divergence output beam, enabling e.g. stand-off detection schemes. Here we report on the use of EC-QCL for the spectroscopic detection of hazardous substances, using stand-off detection of explosives and sensing of hazardous substances in water as two prototypical examples.

Reliable stand-off detection of residues of explosives is still a challenging task. Imaging MIR backscattering spectroscopy has been shown to be a promising technique for non-contact detection of traces of explosives on various surfaces. This technique relies on active imaging with MIR laser illumination at various wavelengths. Recording the backscattered light with a MIR camera at each illumination wavelength, the MIR backscattering spectrum can be extracted from the three-dimensional data set recorded for each point within the laser illuminated area. This way, contaminated areas can be clearly identified on the basis of characteristic finger print backscattering spectra and separated from the corresponding spectra of the underlying material. To achieve a high selectivity, a large spectral coverage is a key requirement. Using a MIR EC-QCL with a tuning range of 300 $cm^{-1}$, different explosives such as TNT, PETN and RDX residing on different background materials, such as painted metal sheets, cloth and polyamide, could be clearly detected and identified. For medium stand-off detection distances (<3 m), concentrations down to some 10 $\mu g/cm^2$ could be detected. For larger concentration of explosives, stand-off detection over distances of up to 28 m could be demonstrated.

Due to their high spectral brightness, EC-QCLs are ideally suited also for the spectroscopic detection of contaminants in water. Due to the high background absorption in water, FTIR spectroscopy is limited to an absorption path length of ~10 µm. Here we realized an EC-QCL based measurement system with an extended single-pass optical path length of 100 µm, which allowed sensitive detection of contaminants in water even when using an uncooled pyroelectric detector. The significantly increased thickness of the water film which can be probed will allow the use of MIR absorption spectroscopy also for in-line process analysis, without the risk of frequent clogging of the measurement cell and without the need to apply high pressures in order to achieve a sufficient flow rate.

Invited

# New gas sensing applications in mid-IR

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Tunable Laser Spectroscopy has become an important tool for process monitoring in industrial applications. These applications require reliable unattended operation with service intervals of several years. This can only be achieved using high quality laser diodes operating CW single-mode at room temperature, presently readily available in the near infrared wavelengths. Until now the lack of suitable laser sources in the main hydrocarbon absorption bands has limited the applicability of TLS in the chemical industry. Mid-infrared type-I quantum well DFB laser diodes with emission wavelengths up to 3.4 μm have recently been developed and successfully utilized in spectroscopic applications [1,2]. DFB interband cascade lasers operating near room temperature with an emission wavelengths covering the wavelength range beyond 3.4 μm have recently been developed by several groups [3,4]. This is of great significance since it will enable real-time monitoring of many important chemicals such as aldehydes, alcohols and hydrocarbons.

Formaldehyde ($H_2CO$) is the most important aldehyde produced commercially. It is broadly 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. Formaldehyde has been classified as carcinogenic to humans by the International Agency for Research on Cancer (IARC) since 2004. California Air Resources Board (CARB) issued regulations in 2009 to limit formaldehyde emissions from composite panels used in finished consumer products. As $H_2CO$ emission standards become more stringent there is a clear need for continuous monitoring of formaldehyde emissions. We have recently demonstrated application of tunable laser spectroscopy (TLS) incorporating a room-temperature distributed feedback interband cascade laser (ICL) for detection of formaldehyde [5]. With the ICL made available to us by the University of Würzburg and nanoplus GmbH we could reach the lines around 3493 nm which has an absorption that is approximately 60% of that of the strongest lines in the band around 3.6 μm. The measured ICL tuning characteristics has shown to be very suitable for the TLS technique.

We have also recently investigated possibilities for remote detection of alcohol vapor against drunk driving utilizing Mid-IR laser sources.

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# From MIR to FIR – physical chemistry vs chemical physics. Terahertz waves in sensing technology.

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One of the research methods of chemistry and physics is spectroscopy. Medium Infra-Red MIR (400-4000 cm-1) and Near Infra-Red NIR (4000-140000 $cm^{-1}$) regions were dominated in the past by Physical Chemistry domain. These regions were interesting because of their ability to determine a molecular structure of the investigated medium. Introduction of the Fourier Transform technique (Fourier Transform Infra-Red – FTIR) made it possible to obtain good quality spectra, especially in the region 400-1600 $cm^{-1}$ (MIR). Still the terahertz region was difficult to investigate. It was even called a “dead land”. Only Raman spectroscopy delivered information about this spectral region. The appearance of the terahertz technique (T ray) allowed the study of intermolecular interactions and provided information about the crystal lattice modes. In that way it became a valuable complement to the X ray technique. Far Infra-Red (THz) region is the main object of interest of Chemical Physics domain. Fig. 1 explains the areas of interests of Physical Chemistry, Chemical Physics, and Terahertz technique as well. The spectral boundaries presented in the figure are blurred and overlap mutually.

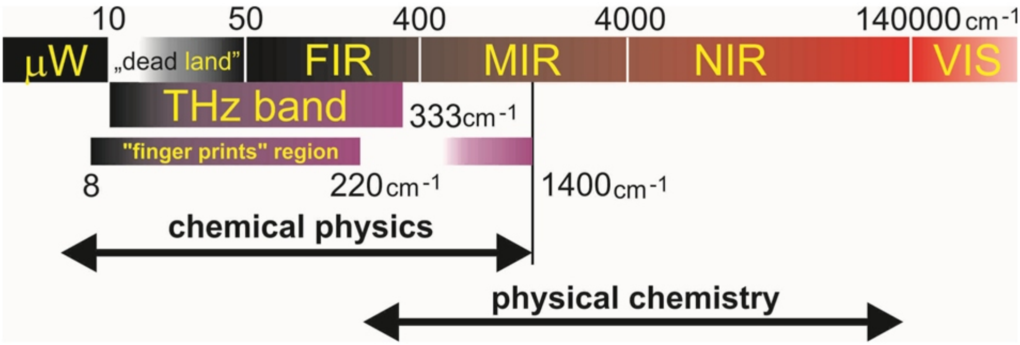


Fig. 1 Higher strip – a traditional picture of a long wave optical electromagnetic spectrum from microwaves (μW) trough far-infrared (FIR) medium-infrared (MIR), near-infrared (NIR), up to visible region (VIS). So called “dead land” is specified. Medium strip – terahertz band (THz) is specified. Lower strip - specified is a “finger prints” region from 8 to 220 $cm^{-1}$ or even to 1400 $cm^{-1}$.

The terahertz technique moved the FIR region below a few reverse centimeters. FIR and THz regions overlap mutually. Very often the terahertz band is located by investigators between 10 and 333 cm-1 (0.3 – 10 THz). It is possible to specify so called a “finger prints” region located approximately from 8 to 220 cm-1. The studied media in the region give the characteristic spectra that allow discriminate the different chemical compounds.

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# Miniaturized TDLS optical gas sensing devices

**Alexandru Popescu[1], Christian Kraeh[1], Harry Hedler[1], Rainer Strzoda[1], Christian Bayer[2], Jörn Stolle[2] and Maximilian Fleischer[1]**

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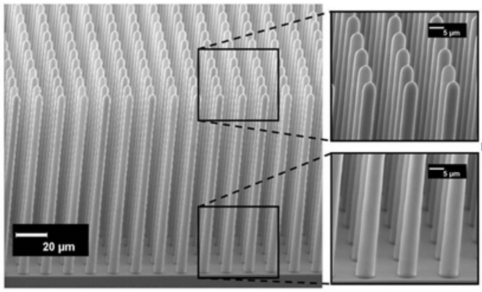


**Fig. 1** Sample SEM picture of poly-Si rods on a Si substrate: The high aspect ratio of roughly 1:25 allows us to treat this sample as a 2D PhC.

Optical gas sensors and in particular tunable diode laser spectroscopy (TDLS) combine high selectivity of target gases with high accuracies. In order to profit from the outstanding advantages of this technique on a widely available basis, compact and integrated devices are needed. Towards such cost effective sensors two key developments are in the current focus of our efforts: (1) The integration of the entire hardware and (2) a reduction of the absorption path length. The integration of the sensor hardware comprises in particular the temperature and current drivers, the signal acquisition and evaluation, as well as the communication module. By employing appli-cation specific integrated circuits a significant form factor and cost reduction will be achieved. Detection limit and accuracy strongly depend on the absorption line. Within the emission spectrum of available laser diodes, only few species exhibit strong absorption lines, which allow short path lengths while keeping the detection limit to a suitable level, e.g. a detection limit of approx. 50 ppm within 1 mm path length can be reached for the $CO_2$ absorption near 2.7 µm. $CO_2$ is one important target for industrial applications, but it is also desirable to access other species with the same integrated device. Besides the change in laser wavelength the required absorption path length has to be considered in order to guarantee satisfying accuracies and detection limits. We explore the employment of photonic crystals (PhCs) as suitable solution. PhCs are able to decrease the group velocity of light by orders of magnitude and thus increase the interaction time of the probe light with the gas by the same factor. Hence, a significant reduction in size results, while assuring the same measurement accuracies. The Photo Assisted Electrochemical Etch technology allows us to manufacture large arrays of Si needle PhCs, cf. Fig. 1. From simulations of band diagrams, we identified the second TE-mode as possible target mode, cf. Fig. 2. Its pronounced flatness near the symmetry point corresponds to low group velocities of the guided light within this mode.

More precisely, by choosing a lattice constant of *a*=3.56 µm and a rod radius of *r*=0.89 µm a normalized frequency of *a*/ =0.46667 near results and matches thus a Methane absorption line at 7.625 µm as our first target. As the group velocity does not extend 0.01 *c* within a bandwidth of approx. 3.5 nm, we can shorten the path length by the same factor. Additionally, the TE modes exhibit pronounced field strengths outside the silicon rods, which increases the interaction of the probe light with the gas sample.

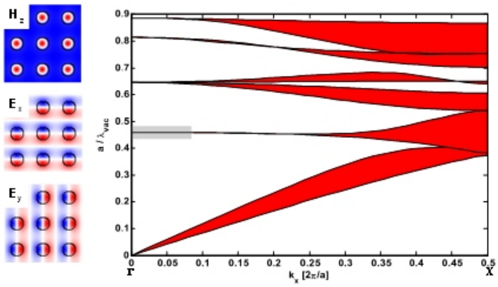


**Fig. 2** Simulated folded band structure of TE modes for a square lattice of silicon rods: The second band can be used for slow light propagation in -X direction, as its pronounced flatness within the shaded area for small wave vectors $\mathbf{k}_X$, corresponds to low group velocities. The mode shows high field strengths outside the silicon rods, which is advantageous for the gas detection scheme.

# Resolving methane isotopologues with a 3.3 µm DFB-diode laser using PAS

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The measurement of hydrocarbon concentrations is essential to many applications from medical diagnostics to natural gas exploration. Spectroscopic analyzers strive to use radiation sources in the region of strongest absorption around 3.3 µm. For many years the source of choice has been the optical parametric oscillator [1]. These OPOs feature a wide spectral tuning range and high optical output power. However, their drawbacks are the high investment costs as well as the bulkiness and complexity of use. Also, they tend to have a relatively broad spectral linewidth compared to other laser technologies. Therefore, a definite need exists for new radiation sources in this wavelength region. Recently, quinary semiconductor materials opened a new path for simple, compact and easy to use lasers [2].

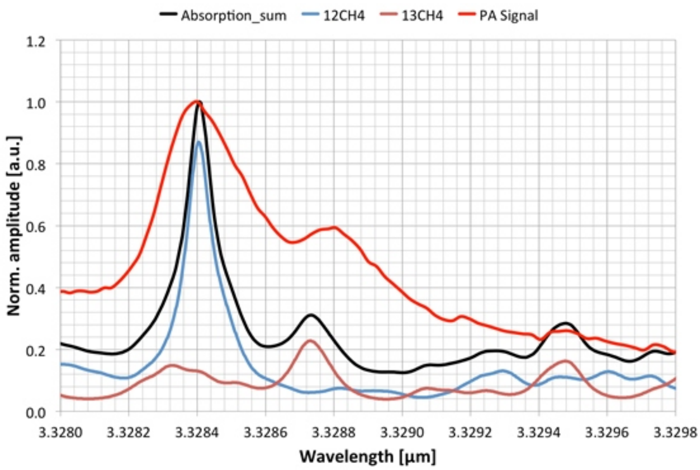


**Fig. 1** Measured spectrum (red) of both methane isotopologues compared to simulated absorption spectra.

We conducted first-time photoacoustic methane measurements resolving both isotopologues (12CH4 and 13CH4) using such a novel DFB-type laser diode. It provides emission wavelengths ranging from 3.328 µm to 3.335 µm. The spectral linewidth is with ca. 0.1 pm around 100 times finer than the absorption structures of interest. As a result, we were able to perform precise measurements of the rotational vibrational transitions of the main isotopologues of methane in the v3 band (Figure 1). The detection limit of our setup lies in the ppb range.

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# Differential optical dispersion spectroscopy for high accuracy trace-gas sensing

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In past decades laser absorption spectroscopy based trace-gas sensors have proven their capability of sensitive and selective chemical detection. However, in some applications current technologies do not always meet demands for higher dynamic range, measurement accuracy and immunity to environmental conditions. These requirements can be addressed through application of molecular dispersion spectroscopy [1]. In this talk a new spectroscopic technique that is based on molecular dispersion measurement and enables new capabilities in trace-gas detection will be discussed.

A differential optical dispersion spectroscopy (DODiS; optical setup shown in Fig. 1a) enables simultaneous and instantaneous measurement of both molecular absorption and molecular dispersion signals originating from two different gas samples. Because the absorption process is cumulative while the dispersion spectrum provides a differential information related to the samples, truly optical addition/subtraction of absorption/dispersion spectra of two different samples can be performed (as demonstrated in Fig. 1b). This is a unique property that is not available with any conventional absorption-based technique. Using DODiS, a single spectral scan can provide information about two unknown samples or if a well-known reference gas mixture is used the differential information can be utilized to a real time system calibration. With these capabilities DODiS can provide a powerful tool for various demanding trace-gas sensing applications including: isotopic ratiometry where a direct comparison of a sample and a reference gas is required, or in environmental/industrial monitoring where unwanted interference from other molecular signatures can be conveniently removed through optical subtraction. The fundamentals of DODiS measurement principle and proof-of-concept experiments demonstrating its potential applications will be presented in details.

The authors would like to acknowledge financial support by the National Science Foundation CAREER award CMMI-0954897 and Dr. Chung-En Zah from Corning Inc. for providing a laser for this study.

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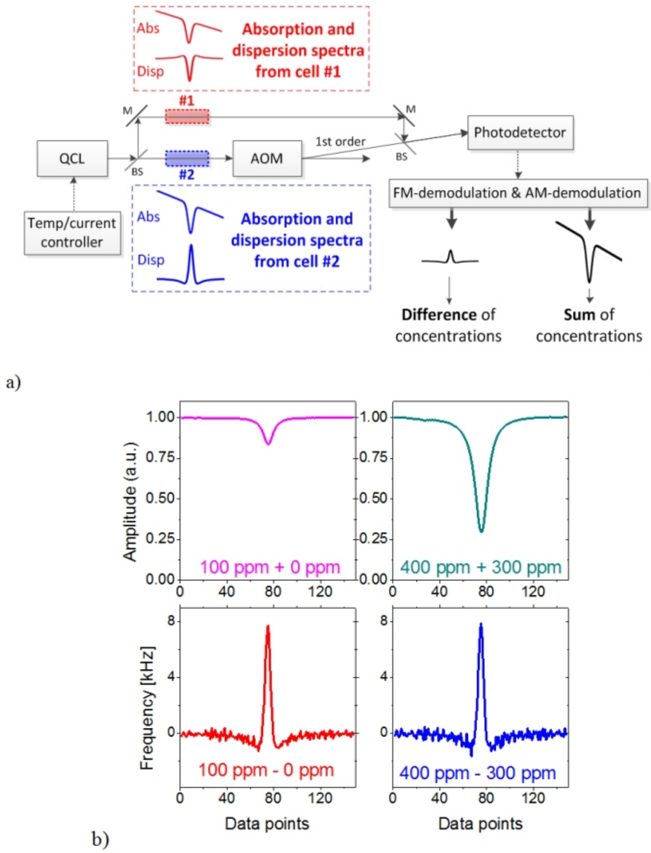


**Fig. 1** a) An optical setup used to perform DODiS (QCL – quantum cascade laser, AOM – acousto-optical modulator, M – mirror, BS – beam splitter). When the laser is chirped, absorption and dispersion spectra of the two samples can be retrieved through AM- and FM-demodulation of the heterodyne beatnote created by two optical waves. Two spectra are recorded simultaneously and effectively provide information about difference and sum of concentrations in two gas cells, from which the individual concentrations can be easily retrieved; b) An example of DODiS spectra (absorption spectra are baseline-corrected) recorded with 4.526 μm QCL when gas mixtures of $N_2O$ in $N_2$ with concentrations of 0 and 100 ppm (left panels) and of 300 and 400 ppm (right panels) were flown through cells #1 and #2 respectively (pressure in both cells was set to 50 Torr). Two dispersion spectra look almost identical in both cases and they indicate the 100 ppm difference in concentrations, while the transmission spectra clearly show increase in absorption governed by exponential Beer-Lamberts Law.

Invited

# Recent progress in mid-infrared laser sources at Center for Quantum Devices, Northwestern University

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Due to concentrated efforts put around wavelengths of 4.6-5  m [1], the wall plug efficiency of InP based mid-IR QCL has been pushed up to 21% in room temperature continuous wave operation with over 5 W output power [2].

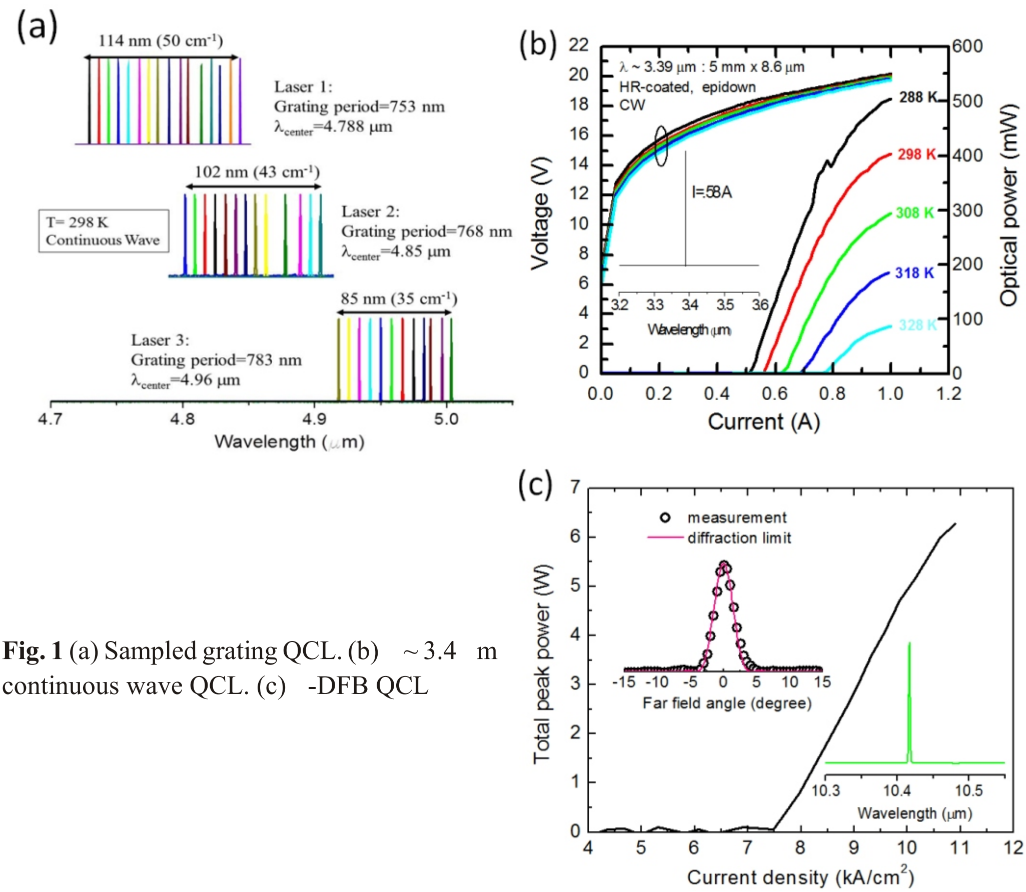


**Fig. 1** (a) Sampled grating QCL. (b)  ~3.4  m continuous wave QCL. (c)  -DFB QCL

With the availability of a highly efficient gain medium, advanced device architectures are explored. Using a sampled grating structure [3], we have significantly increased the tuning capability of a single distributed feedback (DFB) QCL by nearly an order of magnitude. A total of only 3 devices show a gap-free electrical tuning of 120 $cm^{-1}$ with more than 100 mW output power in room temperature continuous wave operation (Figure 1(a)). In addition, the shortest cw wavelength has been dramatically reduced down to 3.4 m with about 500 mW cw power at room temperature (Figure 1(b)). This is accomplished by significantly increase the strain level of the heterostructure, while keeping an overall high material quality [4]. Moreover, we have introduced a new type of broad area devices, called -DFB lasers [5], and demonstrated the highest peak power at wavelengths longer than 10 m, in room temperature single mode operation with diffraction limited beam quality (Figure 1(c)).

The InP based quantum cascade technology has demonstrated its superior capability covering the entire mid-IR wavelength range. Continuous effort is being devoted to all of the above mentioned areas with the intention to create compact laser sources that meet the requirement of different applications.

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Invited

# Single-mode interband cascade lasers for chemical sensing

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The interband cascade laser (ICL), a hybrid of the conventional interband semiconductor laser and the quantum cascade laser geometry, allows access to any mid-infrared (mid-IR) wavelength between 3 and 6   m using the InAs/Ga(In)Sb/AlSb type-II material system.  ICLs differ from any other class of semiconductor laser, conventional or cascaded, in that most of the carriers producing population inversion are generated internally, at semi-metallic interfaces within each stage of the active region. Simulations of the carrier distribution in the active region led to a new design principle of rebalancing the electron and hole densities in the active QWs, with laboratory devices exhibiting input power as low as 29 mW at room temperature and a maximum cw operating temperature of 107 $^{o}$C. This presentation will include a review of the ICL's unique internal carrier generation and transport processes and the latest results for broad-area and narrow-ridge devices, including single-mode distributed-feedback (DFBs) lasers.

Interband cascade lasers with 5 active stages were grown on GaSb substrates and included an active region with the "W" configuration, GaSb/AlSb hole and InAs/AlSb electron injectors, two GaSb separate-confinement layers (SCLs), and InAs:Si/AlSb superlattice claddings. Optimization of the device design was based on the pulsed characterization results for broad-area lasers. Room-temperature thresholds as low as 140 A/cm$^2$ were obtained for emission wavelengths of   = 3.3–3.7 μm, with characteristic temperatures $T_0$ in the 42–50 K range (over the span 300–350 K). Internal losses inferred from the pulsed differential slope efficiencies at room temperature were as low as 5 cm$^{-1}$ in the 3.5–3.8 μm spectral range. Pulsed threshold power densities of 320 W/cm$^2$ at 300 K are ~30 times lower than for the best mid-IR QCLs, although the higher characteristic temperature $T_0$ and larger number of stages of the QCLs allows them to generate higher powers at room temperature.

Narrow ridges of width 5–11 μm were fabricated by photolithography and reactive-ion etching using a Cl-based inductively-coupled plasma process that stopped at the GaSb SCL below the active QWs. The ridges were subsequently cleaned with a phosphoric-acid-based wet etch to minimize damage from the ICP RIE process. The ridges were covered with Au electroplating and mounted in the epi-side-down geometry to improve heat dissipation. An 11 μm x 4 mm ridge with HR/AR coatings generated a cw output power at RT of 291 mW. When the cavity was shortened to 0.5 mm (HR/U facets), the maximum wallplug efficiency at 25$^{o}$C reached ~15%, which compares to the maximum cw wallplug efficiency of 21% for QCLs emitting at = 4.6 μm.

For DFB lasers, ridges were defined in the manner described above except that first-order DFB gratings were realized by patterning high-index Ge layers on top of narrow ridges of the ICL with relatively thin top cladding. Continuous-wave (cw) operation was observed in a single spectral mode ( = 3.7–3.8 μm) at temperatures in the range 20–80 $^{o}$C. One device generates over 27 mW of cw single-mode output at T = 40$^{o}$C, with a side-mode suppression ratio > 30 dB, while at 80 $^{o}$C it still emits > 1 mW. At 20 $^{o}$C, a second device lases in a single spectral mode with < 100 mW of drive power. The maximum tuning range with temperature is 21.5 nm.

# Interband cascade lasers with low threshold current densities

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Within the last decade interband cascade lasers (ICLs) [1] have evolved to an outstanding laser type. While it is able to cover a wide range of the mid infrared region (MIR) [2] independently from the materials bandgap it requires significantly lower threshold currents than comparable quantum cascade lasers or diode lasers. While spatially indirect interband transitions between electron states in the conduction band and hole states in the valence band facilitate laser operation, only electrons are injected and extracted. The special broken gap alignment in the GaSb material system enables electrons to re-enter the conduction band after a radiative interband transition. Early ICL structures suffered from high threshold current densities and low temperature stability. After several design variations concerning the active region as well as the waveguide the laser performance could be gradually improved. Various parameters that have shown a high impact on the laser performance are briefly shown. Via the lately published concept of rebalancing of internally generated carriers [3] the laser characteristics could be even

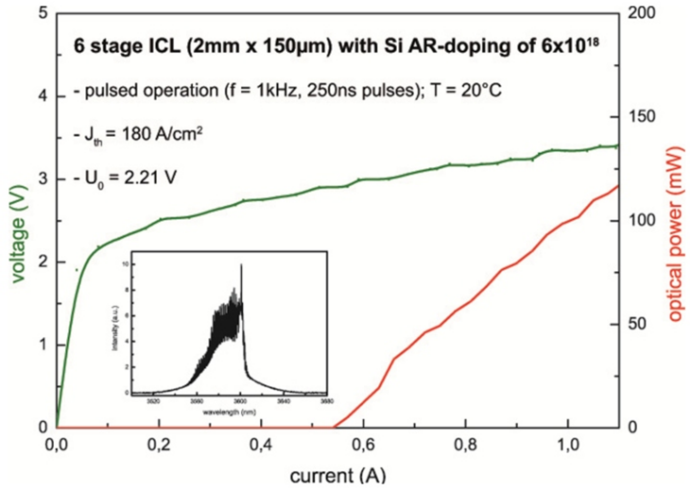


**Fig.** 1 I-U and I-P characteristics of a 2 mm x 150 µm BA-laser.

more improved. By dramatically increasing the doping concentration from $2x10^{17}$ to $6x10^{18}$ in the active region the threshold current density for 150 µm wide and 2 mm long broad area lasers with an emission peak at 3.6 µm could be improved from 380 A/cm$^2$ to only 180 A/cm$^2$ at room temperature (see fig. 1). These results as well as room temperature continuous wave operating lasers will be presented.

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# Time-resolved spectral behavior of ring cavity surface emitting QCLs (RCSE-QCLs)

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Quantum cascade lasers (QCLs) are considered as reliable sources of coherent light ranging from the mid-infrared (MIR) to the terahertz spectral region due to their constant improvement. Recently single-mode operation of a new type of ring cavity surface emitting QCLs (RCSE-QCLs) was demonstrated by Mujagic et al. [1].

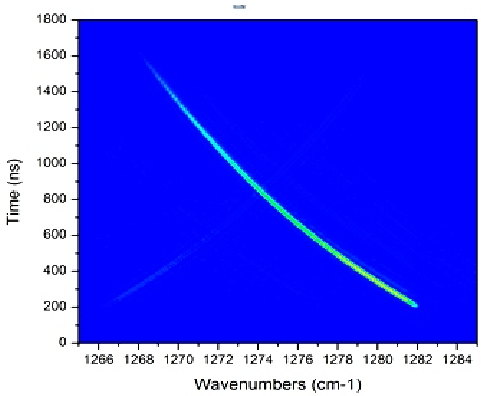


**Fig. 1** Time-resolved RCSE-QCL emission for a 2 µs pulse and 40 kHz repetition rate.

This design of a facetless ring shaped resonator with a second order grating on top combined with the large overall emission surface leads to reduced threshold currents, enhanced optical power output and a highly reduced beam divergence as compared to conventional facet emitters in distributed feedback (DFB) or Fabry-Pérot (FP) configuration [2]. These novel properties render the RCSE-QCLs ideal for gas sensing applications.

In this work the time resolved down-chirp spectral behavior of RCSE-QCLs in the 1235–1285 cm$^{-1}$ spectral range was investigated using a step-scan FT-IR spectrometer (Bruker Vertex 80v) with 2 ns temporal and 0.1 cm$^{-1}$ spectral resolution. The optical power output was measured in pulse-mode operation with pulse lengths of 0.1–2 µs and repetition rates of 5–500 kHz at different temperatures, Fig. 1 shows an example. Furthermore, the RCSE-QCL emission was compared with the emission characteristics of standard DFB type facet emitters.

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# High-power GaAs/AlGaAs quantum-cascade lasers

**Kamil Kosiel, Anna Szerling, Piotr Gutowski, Piotr Karbownik, Anna Wójcik-Jedlińska, Justyna Kubacka-Traczyk, Iwona Sankowska, Kamil Pierściński, Dorota Pierścińska, Emilia Pruszyńska-Karbownik, Maciej Bugajski**

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The presentation concerns the $Al_{0.45}Ga_{0.55}As$/GaAs quantum-cascade lasers, which technology has been studied by us for the last few years [1–3]. Though the general design of this high-barrier heterostructure, based on 3QW diagonal-anticrossed scheme, comes originally from the work presented already in 2001 [4], it seems there is still room for improvement of the optical power generated by it. So far, we have obtained up to 6W output peak powers generated by our lasers at cryogenic conditions (77K), and corresponding ~50mW powers at the room temperature (300K). The cryogenic efficiencies for lasers with metallic-coated high-reflectivity (HR) back mirrors are up to ~1W/A.

The laser heterostructures were prepared by molecular-beam epitaxy technique, by use of multi-step interrupted-growth method specially designed in order to maximize the stability of the overall epitaxial conditions. This method, however, was applied in order to overcome the MBE system limitations, rather, than for the further improvement of the state-of-the-art lasers' output power. Particularly important was the optimization of doping profile throughout the structure, as well as appropriate V/III ratio for the growth of different regions of the laser. The chip fabrication comprised of wet mesa etching, electrical insulation by use of $Si_3N_4$ and Ni/AuGe/Ni/Au metallization followed by thinning the wafer down to 100 m and further bottom metallization by AuGe/Ni/Au [2,5]. Back-mirror golden HR coatings were prepared with special care, that was crucial for lasers' increased efficiency.

The lasers were successfully applied in prototype trace-ammonia detection system working with ppb-level resolution and with 73ppb sensitivity (for 95% transmission). There is still room for development of that system, however, for example by use of the multipass cell with longer optical path (the used one was effectively 36m-long). The system may be applied for detection of other types of gases, as well [6].

Recently the lasers have been successfully tested in the laboratory free-space data link system.

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# Specialized GaSb based laser sources for sensing: Devices offering an extended tuning range and Devices with high optical output power

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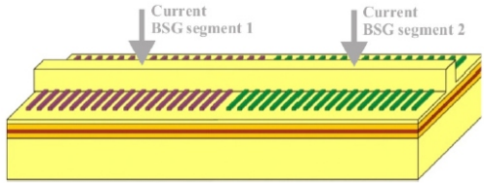
Tunable diode laser spectroscopy (TDLS) is a versatile and very successful approach for high performance gas sensing. Especially in the wavelength range between 2.0 and 3.5 µm there is a wide variety and a growing number of applications, since the absorption features of many technologically and industrially important gas species are located in this range [1-3]. A crucial requirement for TDLS is the availability of spectrally monomode emitters such a distributed feedback (DFB) lasers. The GaSb system has become the material system of choice for processing laser diodes in the wavelength range of interest between 2.0 and 3.5 µm.



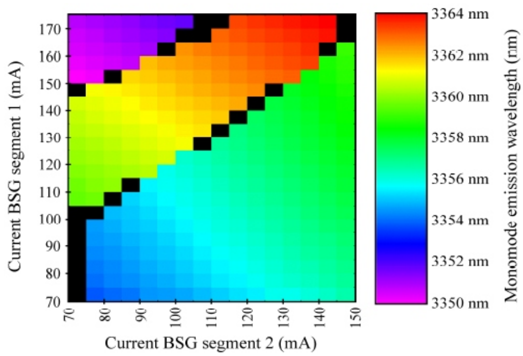




**Fig. 1** Schematic design of a widely tunable laser (two-section laser with binary superimposed gratings) and mode map of a fabricated device in the 3.35 µm wavelength range.

In this talk, we will present devices with performance figures surpassing regular DFB lasers with respect to tuning range or laser output power. Device fabrication uses overgrowth-free processing routes having significant advantages for GaSb based heterostructures, for which an overgrowth is extremely hard to achieve due to their reactivity with oxygen.

While the current induced tuning range of a DFB laser is typically limited to a few nanometers, a number of applications would benefit from monomode laser diodes with an extended tuning range. These include liquid sensing as well as multi-component gas analysis. To enable such applications, monolithic widely tunable devices were investigated in the wavelength range of interest. Figure 1 schematically depicts the design of a fabricated two-section device based on binary superimposed grating structures. Widely tunable device operation has been obtained by independently injecting two currents into the two laser sections. A corresponding mode map (with the emission wavelength color-coded for the respective combination of currents) is also shown in Figure 1.

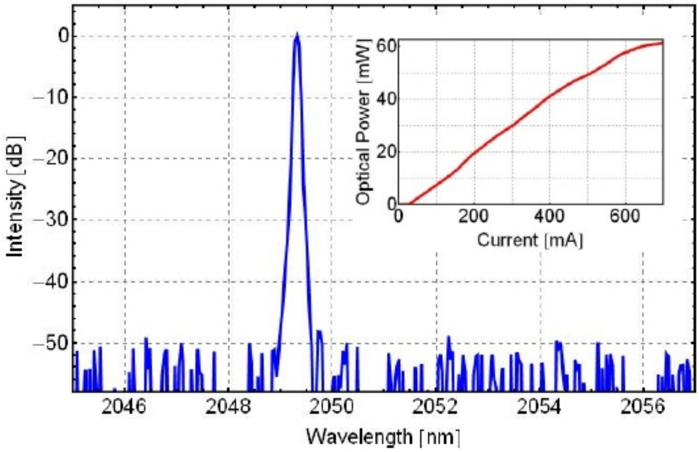



**Fig. 2** Monomode spectrum and L-I curve (inset) of a fabricated 2.05 μm device with output power >50 mW in CW operation.

While for most application in gas sensing an optical output power < 10 mW is more than sufficient, certain applications (e.g. in dust carrying environments or over long distances) would benefit from higher laser output powers. Figure 2 shows a GaSb based device example in the 2.05 μm wavelength region offering an output power > 50 mW.

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Invited

# GaSb-based structures for mid-infrared emitting lasers: optical study

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Applications related to the sensing of hazardous and environmentally relevant gasses, especially by the optical manner, drive the growing demands with respect to all the necessary components, requiring cheap, compact and fast laser sources. However, in many cases the respective devices able to emit in the ranges corresponding to the maximal absorption, which for many gasses falls into the mid infrared range, and which would additionally offer a continues wave and single mode operation at room temperature, are not commercially available. As most of the gasses, for instance hydrocarbons, occur in a mixture or a background of other gaseous chemicals, multiwavelength or widely tunable lasers are very often required for a proper detection and full functionality. One of the possible routes is constructing the semiconductor lasers exploiting multinary compound materials based on GaSb. The target range, which is usually about 3-4 μm, and beyond into the longer wavelengths, can be reached by using several approaches, starting from short wavelength quantum cascade lasers, through common type I quantum-well-based laser diodes, up to the so called interband cascade laser, a solution being a hybrid of the two former ones and employing a combination of materials making a broken gap system and a type II indirect in the real space optical transition as the active one.

There will be reviewed the optical properties of such kind of active regions with a special focus on the InAs/InGaAsSb type II QWs grown on GaSb substrates, and the related current challenges with respect to the device characteristics. This will cover such issues as the band offsets importance, and its sensitivity to the layers composition, the active type II transition oscillator strength versus various structure parameters and external factors as temperature or electric field, and the predominating carrier loss mechanisms. For that a combination of several spectroscopic techniques have been used, both emission-like (photoluminescence) and absorption-like (modulated reflectivity spectroscopy) supported by the energy level calculations employing a multiband **kp** model. Eventually, the potential for further material optimization and prospects for the improved device performances will be given.

# Dilute nitride InAsN multi-quantum wells on metamorphic buffer layer on InP for mid-infrared optoelectronics

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Multi-quantum wells (MQWs) on InP substrates have been successful in telecom lasers operating at wavelength of 1.55 um. In order to realize lasers on InP operating in the important mid-infrared (MIR) spectral range (3-5 m), a new class of alloy – dilute nitride has been employed as quantum wells where the nitrogen incorporation into InAs reduces the bandgap energy and simultaneously reduces the compressive strain. Such solution successively led to lasers on InP operating at wavelength of 2.6 m. However, longer wavelength from such structure is extremely challenging due to the significant compressive strain and the severe material quality degradation while more nitrogen is incorporated. In this paper, we present a solution towards MIR lasers on InP by combining metamorphic buffer layer (MBL) with our previous success in epitaxial growth of high quality InAsN epilayers. InAsN MQWs on indium-rich InGaAs MBL on InP have been successfully grown, where bright room temperature photoluminescence (PL) at wavelength of 3.0 m was revealed.

The MBL structure consists of a 5 m-thick InPSb graded buffer layer followed by a 20 nm-thick $In_{0.73}Ga_{0.27}As$ layer, where were grown by metal organic vapour phase epitaxy (MOVPE) on (100) InP substrates. Then 5 InAsN/InGaAs MQWs (5nm/25nm) were grown on this MBL layer by plasmaassisted molecular beam epitaxy (PAMBE). The nitrogen composition was controlled by precalibrated growth conditions to maintain a high quality of the QWs. Growth temperature was varied between 460–520 $^{o}$C with a growth rate of 1.0 μm/h and a fixed nitrogen plasma setting (power of 160W and a nitrogen flux of $5.0 \times 10^{-7}$ mbar). All of the growths were performed under the minimum As flux to maintain an efficient nitrogen incorporation. The resulting InAsN MQWs structures were extensively studied by 4-300 K photoluminescence (PL) with an $Ar^{+}$ ion laser (514 nm) and photoreflectance (PR) measurements.

Fig. 1 shows the 4K photoluminescence from the samples grown at different temperatures. It reveals that the emission peak energy, line-width and intensity are sensitive to the growth temperature. Reducing growth temperature yields a reduced emission energy, increased peak intensity and narrower line-width. More important, the PL emission persists up to room temperature for the samples grown at temperatures below 460°C. These observations indicate that growth temperature is the key factor in producing device quality InAsN MQWs on MBL on InP. To investigate the mechanism of the behavior, temperature varied PL, laser excitation power varied PL and PR measurements were performed. The experimental results and the origin of the strong PL emission will be discussed in details. This work indicates the promising device applications of dilute nitride MQWs on InP in MIR laser operating at a technological gap of 2.5 – 3.2 m.

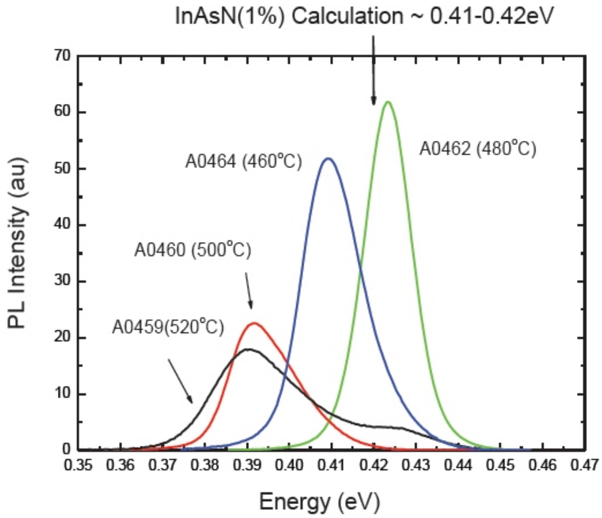


**Fig. 1** 4K PL emission spectra from the samples of InAsN MQWs on InP

# Developments in Fourier-transform-based optical characterization of mid to far infrared semiconductor materials

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Modulation spectroscopy, due to its absorption and differential character, is an excellent tool to study the energies of optical transitions (including the excited state ones) in quantum well (QW) and quantum dots as well as energies of bulk-like QW barriers or intermediate layers. Such experiments have already been successfully employed to study GaSb-based QWs for infrared applications up to 3 μm [1] and including type II structures for even longer wavelengths [2]. Nevertheless, the standard modulation spectroscopy based on diffraction grating monochromators has some limitations due to several reasons like e.g. less sensitive detectors or less efficiency of the probing light sources in case of mid and far infrared (comparing e.g. to the tools used in the visible range) [3]. Due to a growing interest and necessity to investigate structures designed for operation in mid and far infrared range (as infrared detectors, quantum cascade lasers, etc.) it has been proposed to exploit modulation spectroscopy realized by using Fourier-transform spectrometer [3,4]. This approach has already been used to investigate mainly the bulk-like materials and layers. In this work, we demonstrate the application of FTIR modulation spectroscopy (called FTPR) for the investigation of low-dimensional structures for mid infrared spectral region even beyond 5 μm [5]. In addition, we introduce a new experimental approach called Fast Differential Reflectance (FDR) [6]. This technique allows recording the photoreflectance-like spectra similar to those obtained in conventional measurements utilizing the lock-in amplification technique. In FDR approach however, the time of collecting the spectra can be reduced from hours to seconds in comparison to standard grating-based method. Its functionality and comparison to FTPR and FTPL will be demonstrated for HgCdTe layers in the range of 5-15 μm [7], GaInAsSb layers on InAs [8], and long wavelength type type II GaSb/AlSb/InAs/GaInSb/InAs/AlSb/GaSb QWs [9]. Eventually, the future prospects on further extensions to longer wavelength up to the terahertz range will be discussed.

This work was partially supported by the National Science Center within the Grant DEC 2011/03/D/ST3/02640.

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# Optical properties of InGaAsSb/InAs solid solution heterostructures grown by liquid phase epitaxy

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Mid-infrared semiconductor lasers are continuously increasing their application range during the last years including for instance gas sensing for detection and control of the presence or concentration of harmful gases like $CO_2$, $SO_x$, $NH_3$, and many others. There are many concepts of obtaining the laser emission in the range above 3 µm including quantum cascade lasers (QCL), interband cascade lasers (ICL) and lasers based on type I quantum wells. Generally, it is necessary not only to achieve the target wavelength, but also to obtain the room temperature single mode operation, which brings the demands with respect to the active material even further.

A very important parameter, which must be taken into account in designing the laser structures, is the spin-orbit splitting in the valence band. The resonance between the split-off valence bands and the energy of the emitted photons results in a significant increase of the Auger recombination rate and enhanced intervalence band absorption, which deteriorates the luminescent characteristics of the IR-emitters operating at the 3 - 5 µm.

In this work, we investigate thick layers of narrow-gap InGaAsSb material with high In content grown on InAs substrates by liquid phase epitaxy. This type I quantum well structures are potentially able to cover spectrally the range far above 3 µm.

We present photoluminescence (PL) and photoreflectance (PR) study of temperature dependence of the energy gap and the spin-orbit split off transition in $Ga_{0.84}In_{0.16}As_{0.22}Sb_{0.78}/In_{0.83}Ga_{0.14}As_{0.82}Sb_{0.17}/InAs$ layers measured by the FTIR spectrometer in the mid infrared range. The energy gap thermal coefficient has been found to be  = -0.43 meV/K. Additionally, conventional photoreflectance spectra have been measured in the vicinity of the the spin-orbit split-off transition versus temperature. For the investigated $In_{0.86}Ga_{0.14}As_{0.83}Sb_{0.17}$ alloy, the spltiting has been determined to so = 0.460eV and shown that this energy is temperature insensitive. These results demonstrate that there the possibility of tuning the temperature dependence of the fundamental gap energy of the narrow-gap solid solution independently of the spin-orbit splitting [1], which is a crucial property in optimizing the structure for high perforamcne mid infrared lasers.

This work was supported in part by the RBRF grant #11-02-00234 and programs of Presidium of RAS and Department of Physics Sciences of RAS. Additionally, we would also like to acknowledge the Ministry of Science and Higher Education for financial support from the Iuventus Plus Program.

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Invited

# InAs-based interband cascade lasers

**Rui Q. Yang[1], Lu Li[1], Zhaobing Tian[1], Yuchao Jiang[1], Lihua Zhao[1], Robert T. Hinkey[1,2], Hao Ye[1], Tetsuya D. Mishima[2], Michael B. Santos[2], Joel C. Keay[2], Matthew B. Johnson[2] and Kamjou Mansour[3]**

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Taking advantages of type-II band-edge alignment in InAs/GaSb/AlSb material system, interband cascade (IC) lasers [1] reuse every injected electron to generate multiple photons with high quantum efficiencies and can cover entire mid-infrared spectrum. In contrast to quantum cascade lasers based on intersubband transitions, IC lasers circumvent the fast phonon scattering issue by using the transition between conduction and valence bands for photon emission. As such, the threshold current density is significantly lowered with high voltage efficiency, resulting low power consumption. The exploration and development of IC lasers over the last 16 years has proven quite successful. Lasing in continuous wave operation at room temperature and above has been demonstrated for a wide wavelength range of 2.9 to 5.7 m [2,3]. Here, we present our recent progress in InAs-based IC lasers [4–7]. In these lasers, we replace the InAs/AlSb superlattice cladding that is typically used for GaSb-based IC lasers with a highly doped InAs cladding layer. Using this plasmon-waveguide approach, we have improved the thermal dissipation and index-contrast of the cladding layer with the cascade stages. This has enabled the extension of IC lasers to long wavelengths up to 10.4 μm. We will also report our recent IC lasers with simultaneous lasing at two different wavelengths in a single waveguide, and other IC lasers with wide tuning ranges.

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Invited

# Single mode operation of monolithic GaSb VCSELs

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Electrically-pumped GaSb-based Vertical Cavity Surface Emitting Lasers (EP-VCSELs) intrinsically exhibit good characteristics for gas sensing such as single-mode operation, low threshold current and continuous-wave (CW) operation in the mid-IR range.

Recently, *hybrid* Sb-Based EP-VCSELs emitting up to 2.6 µm have been demonstrated which operate CW up to 50°C [1]. Efficient current confinement is achieved thanks to a buried Tunnel Junction (TJ). This technology however relies on complex processing steps, including epitaxial regrowth which deteriorates the active zone. An alternative monolithic approach was also developed which uses two n-type DBRs and a TJ. Room temperature operation has been demonstrated in CW mode at 2.3 µm [2] and pulsed mode up to 2.63 µm [3], the record wavelength for III-V-VCSELs to date. However, these devices were based on large-area etched mesas and lacked an efficient electro-optical confinement, leading to high threshold and multimode emission.

In this communication, we report on the fabrication and the performance of monolithic GaSb-based EP-VCSELs with a TJ selectively etched to form an air gap aperture [4]. The monolithic structure has been grown by molecular-beam epitaxy in a single growth run. A total VCSEL thickness of ~14 µm results in ~16 hours growth duration. Devices with 35 µm pillar diameter were processed resulting in EP-VCSELs with 6µm TJ effective diameter.

Typical light-current-voltage (L-I-V) characteristics in CW at various temperatures show that CW operation is obtained up to 70°C. The threshold reaches a minimum value around 1.9 mA at 30 °C which is the temperature at which the detuning between the gain and the microcavity resonance allows the lower threshold.

Single-mode emission is achieved with a Side Mode Suppression Ratio (SMSR) around 25 dB in the whole range of driving currents and temperatures. The electro-thermal tunability allows shifting continuously the wavelength on a 14 nm range with an emission that remains single-mode in the whole range of currents and temperatures [5]. This large continuous wavelength tuning with single mode emission demonstrates that this device is well suited to scan several gas absorption lines as requested for TDLAS applications.

Our work shows that monolithic GaSb-VCSELs represent a viable technology for the development of mid-IR photonic devices and systems.

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# Single mode quantum cascade lasers with shallow-etched distributed Bragg reflector

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Trace gas sensing in the mid-infrared is primarily conducted with quantum cascade lasers (QCLs) utilizing an integrated distributed feedback (DFB) grating to ensure single-mode emission. Since these lasers can only be tuned over a few wavenumbers via temperature tuning on standard Peltier-coolers, different concepts to extend the single-mode tuning range of quantum cascade lasers - e.g for the detection of multiple gas-species - have been pursued: Very broad tuning ranges of up to a few hundred wavenumbers have been demonstrated with external cavity lasers [1]. Since they however require external optics and therefore precise alignment and a stable environment, approaches yielding monolithic devices are vividly investigated. Laser arrays covering more than 100 $cm^{-1}$ have also been realized [2], however since they emit from multiple facets beam combination is necessary and in case of vertically emitting arrays a non-Gaussian far-field is observed [3], which makes beam conditioning rather challenging.

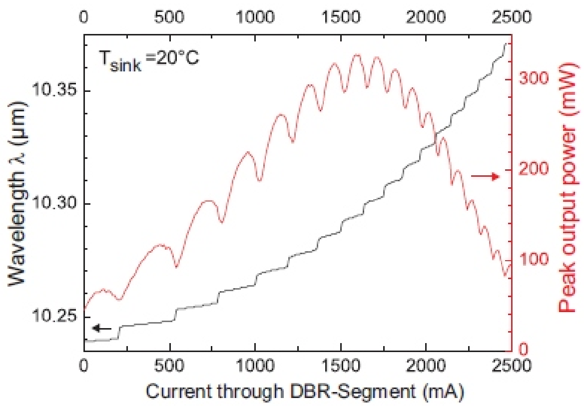


**Fig. 1** Tuning behavior and output power of a DBR-QCL emitting around 10.3 µm as a function of DBR-current at constant heat sink temperature of 20°C.

In order to develop industry-grade devices which are more compact than external cavity lasers and less sensitive to environmental constraints at industrial sites we investigated monolithic quantum cascade lasers which utilize a shallow-etched distributed Bragg reflector (DBR) for wavelength-selective feedback [4]. In contrast to monolithically integrated arrays of DFB-QCLs, a single DBR-QCL device exhibits tunable emission out of a single facet which makes fiber coupling simple and eliminates the need for beam combining.

In this contribution we present DBR-QCLs emitting around 10.3 µm and 13.5 µm with a maximum peak output power of 600 mW and a maximum tuning range of ~ 35 $cm^{-1}$. The characteristic tuning behaviour and output power of a 4 mm long device emitting at 10.3 µm is depicted in figure 2. A detailed analysis of the electro-optic properties and the spectral tuning behaviour will be given. Furthermore the reduced frequency-down chirp as well as the dynamic tuning capability will be discussed. The characteristics of these devices allow for faster tuning and spectrally higher resolved absorption measurements as compared to pulsed DFB devices. Application to sensing of widely spaced gas absorption lines is reported, showing the potential of DBR-QCLs for tunable laser absorption spectroscopy.

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# InAs-based interband cascade lasers for emission around 6 μm

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Interband-cascade-lasers (ICLs) have emerged as efficient laser sources in the mid-infrared in recent years, with the upper wavelength limit at cw room temperature operation being pushed to a current value of 5.6 μm [1,2]. While the underlying concept of type-II transitions in the active region easily allows extending the wavelength beyond 6 μm, the InAs/AlSb superlattice claddings used in GaSb-based ICLs do impose certain challenges. The poor thermal conductivity of these layers combined with the thicker claddings required for longer wavelengths increasingly limits the heat dissipation and hence the device performance [3]. To overcome these limitations, plasmon enhanced claddings on InAs substrates can be used, where the superlattice is replaced by a highly doped InAs layer [4]. As the wavelength comes into range of the plasmon frequency in these layers, the refractive index is drastically reduced, leading to a strong optical confinement.

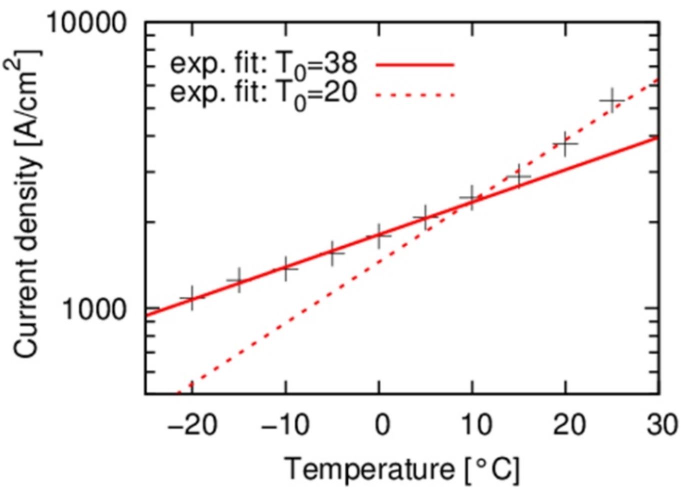


**Fig. 1** Threshold current density vs. temperature for a 1 mm x 0.1 mm shallowly etched broad area device.

In this work InAs-based ICLs with emission wavelengths around 6 µm have been fabricated and characterized. Use of shortened cascades to increase the mode intensity in the active region [5], as well as split hole extractors with two GaSb quantum-wells to suppress possible hole leakage [6] considerably improve device performance. Shallowly etched broad area devices show a maximum operation temperature of 25 °C in pulsed mode, with a threshold current density of 3.7 kA/cm$^2$ at 20 °C (figure 1). Deeply etched structures of the same wafer yield a reduced threshold current density of 2.2 kA/cm$^2$ at the same temperature.

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# Mid infrared fiber lasers

**L. Sójka[1,2], Z. Tang[2], H. Zhu[2], E. Bereś-Pawlik[1], D. Furniss[2], A.B. Seddon[2], T. M. Benson[2] and S. Sujecki[2]**

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Lanthanides have many transitions in the mid infrared wavelength range. However, these transitions are quenched by most of the host materials. Hence, one of the main problems in construction of mid- infrared fiber lasers is founding a proper host material with a low photon energy.

One of the promising materials for the construction of mid-infrared fiber lasers is the chalcogenide glass. The chalcogenide glass has low phonon energy (below 400 $cm^{-1}$) when compared with standard materials used to produce fiber lasers, i.e. ZBLAN and silica glass [1]. It was demonstrated in the literature that the rare-earth doped chalcogenide glass has all necessary parameters to construct an efficient laser i.e: high cross section, long upper lifetimes, and high quantum efficiency of relevant optical transitions [1].

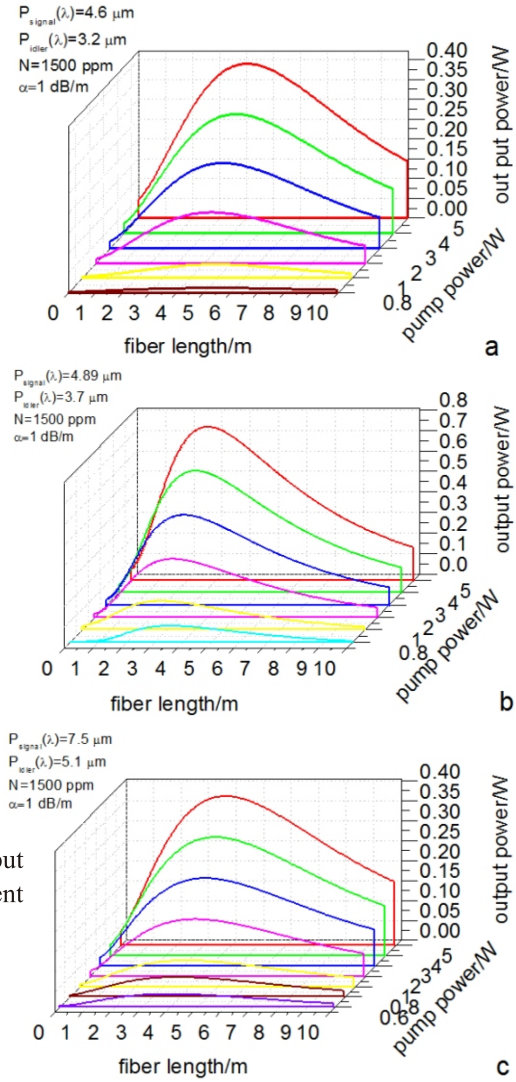


**Fig. 1** Calculated dependence of the output signal power on the fibre length with different pump power for a) $Dy^{3+}$ b) $Pr^{3+}$ c) $Tb^{3+}$.

We present a comprehensive study of chalcogenide glass fiber lasers doped with $Dy^{3+}$, $Pr^{3+}$ or $Tb^{3+}$ that operate in the mid-infrared wavelength range. A set of chalcogenide glass samples doped with different concentrations of rare earth ions was fabricated. The modeling parameters are directly extracted from FTIR absorption measurements performed on the fabricated bulk glass samples using Judd-Ofelt, Füchtbauer–Ladenburg and McCumber theory.

Fig.1 shows results obtained from modeling chalcogenide fiber laser doped with $Dy^{3+}$ , $Pr^{3+}$ or $Tb^{3+}$. These results indicate that an efficient laser action at 4.6 μm, 4.89 μm and 7.5 μm with pumping respectively at 1.71 μm, 2.04 μm and 2.95 μm for all the dopants studied is possible if the fiber losses are maintained at the level of 1 dB/m [2,3].

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Invited

# Mid-infrared semiconductor laser based trace gas sensor technologies: Recent advances and applications

**Frank K. Tittel[1], Rafal Lewicki[1], Mohammad Jahjah[1], Yufei Ma[1], Karol Krzempek[2], Przemysław Stefański[2], Stephen So[3], David Thomazy[3], Johannes P. Waclawek[4], Engelene t.H. Chrysostom[4] and Bernhard Lendl[4]**

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This talk will focus on recent advances in the development of sensors based on infrared semiconductor lasers for the detection, quantification and monitoring of trace gas species and their applications in atmospheric chemistry and industrial process control. The development of compact trace gas sensors, in particular based on quantum cascade (QC), interband cascade (IC) lasers, as well as traditional laser diodes permit the targeting of strong fundamental rotational-vibrational transitions in the mid-infrared, that are one to two orders of magnitude more intense than overtone transitions in the near infrared.

The architecture and performance of four sensitive, selective and real-time gas sensor systems based on mid-infrared semiconductor lasers will be described [1]. High detection sensitivity at ppbv and sub-ppbv concentration levels requires sensitivity enhancement schemes such as tunable laser diode absorption spectroscopy (TDLAS) [2, 3] and wavelength modulation spectroscopy (WMS), photo-acoustic absorption spectroscopy (PAS) or quartz-enhanced-PAS (QEPAS) [2-4]. These spectroscopic methods can achieve minimum detectable absorption losses in the range from $10^{-8}$ to $10^{-11}$ cm$^{-1}$/ Hz.

TDLAS was performed using an ultra-compact, innovative multi-pass gas cell with an effective optical path length of 57.6 m capable of 459 passes between two spherical mirrors separated by 12.5 cm. A 3.36 µm CW TEC DFB GaSb based laser diode operating at 9.5 °C was used as the excitation source [5-7]. For an interference free $C_2H_6$ absorption line located at 2976.8 cm$^{-1}$ a minimum detection limit of 130 pptv with a 1 s. acquisition time was achieved. A new state-of-the-art integrated electronic control and data acquisition module was implemented that allowed further significant size reduction without loss of sensor performance.

A QEPAS based sensor capable of ppbv level detection of CO, a major air pollutant, was developed. We used a 4.61 µm high power CW DFB QCL that emits a maximum optical power of more than 1W in a continuous-wave (CW) operating mode

[5,6]. For the R6 CO line, located at 2169.2 $cm^{-1}$, a noise-equivalent sensitivity (NES, 1 ) of 2 ppbv was achieved at atmospheric pressure with a 1 s. acquisition time. Furthermore, high performance (> 100 mW) 5.26 µm and 7.24 µm CW TEC DFB-QCL (mounted in a high heat load (HHL) package) based QEPAS sensors for atmospheric NO and $SO_2$ detection will be reported [8, 9]. A 1 minimum detection limit of 3 ppb and 100 ppb was achieved for a sampling time of 1 s. using interference free NO and $SO_2$ absorption lines located at 1900.08 $cm^{-1}$ [10,11] and 1380.94 $cm^{-1}$ respectively [12].

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Invited

# Potentials of mid-infrared spectroscopy in emission control

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In more and more countries the emission of nitrogen oxides ($NO_x$) and other industrial gases into ambient air has to be measured permanently due to environmental protection regulations.

At the moment the measurement of nitric oxide (NO) as well as other environmental relevant gases can be performed among others by complex extractive systems (like FTIR or chemiluminescence instruments) or by tunable diode laser absorption spectrometers (TDLAS). Since many customers prefer in-situ measurements, NIR TDLAS spectrometers have become very popular for emission monitoring. Unfortunately some important gases (like NO) possess only weak overtone and combination absorption lines in the NIR and therefore NIR NO spectrometers have only a low sensitivity. The fundamental absorption bands in the mid-infrared spectral region are orders of magnitude stronger; for example, the best absorption line in the 5.2µm-band of NO is approximately 750 times stronger than the best line in the 1.8µm-band.

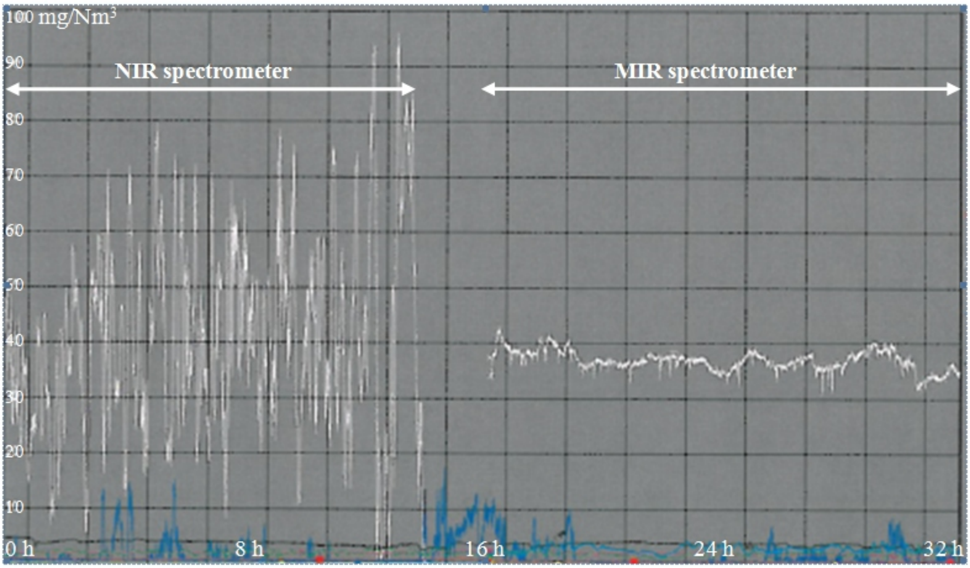


**Fig. 1** Measurement of NO on a stack: On the left hand side with a NIR spectrometer and on the right hand side with a QCL-based MIR spectrometer.

Software simulations based on HITRAN and HITEMP databases have led to a selection of interference-free NO absorption lines in a typical industrial gas mixture. In cooperation with *Corning, Inc*. a quantum cascade laser chip for one of these absorption lines has been prepared and mounted into a HHL package. The laser source has been integrated into a spectrometer which has been tested at an industrial site where it replaced an existing NIR TDLAS spectrometer. Figure 1 shows on the left hand side (white trace) the measured NO concentration with the NIR spectrometer and on the right hand side with the MIR spectrometer ($L$ = 35 cm; $T$ = RT; $p$ = 1 atm). The comparison shows the improvement in sensitivity and stability, and therefore the great potential of QCL-based MIR instruments.

Nevertheless, there are still open issues (e.g. price and availability of QCLs) which have to be solved before MIR spectrometers can be established in industrial markets.

# Applying optical absorption techniques in the deep sea - Underwater greenhouse gas measurements

**Peer Fietzek**

*CONTROS Systems & Solutions GmbH, Kiel, Germany*
*GEOMAR | Helmholtz Centre for Ocean Research, Chemical Oceanography, Kiel, Germany*

Various fields of application demand for the measurement of dissolved greenhouse gases such as carbon dioxide ($CO_2$) and methane ($CH_4$) in rivers, lakes and oceans. Expectably these measurements are often scientifically driven. Investigations of the gas exchange at the air-water interface especially against the background of the increased carbon uptake by the oceans as a consequence to the rising atmospheric $CO_2$ concentrations is just one scientific example; the determination of carbon reservoir contents of deeper water masses as one facet of marine carbon cycle investigations is another. Recently the topic of *ocean acidification* with its potentially severe impact on marine organisms gained a considerable amount of scientific attention. This bold notion refers to the particular property of the aquatic carbonate system that an increase in dissolved $CO_2$ concentration goes along with a decrease in the water's pH. Industrial fields of application cover e.g. measurements within the beverage industry, fish farming and aquaculture as well as within carbon, capture and storage (CCS) projects in which the underwater sequestration of $CO_2$ requires for adequate monitoring. In the case of dissolved $CH_4$ measurements the major field of application is found in the oil and gas sector. Early leak detection investigations at underwater pipelines and long-term monitoring installations at subsea offshore structures are two examples expressing the importance of adequate instrumentation. Furthermore there is a scientific interest in background CH4 concentration monitoring conducted around natural gas seeps and during gas hydrate reservoir investigations as well as in the limnological environment to estimate greenhouse gas contributions of damned waters to the atmosphere.

Within underwater instruments for the measurement of dissolved gases a semi-permeable membrane can be used to separate the liquid on the outside of a device from an internal gas phase. The gases within such a headspace achieve partial pressure equilibrium with respect to their dissolved concentration in front of the membrane. The gas concentration of a target molecule within the gas phase can hence be measured by a favorable technique and the amount of dissolved gas can be derived.

Non-dispersive infrared (NDIR) spectrometry is a proven method for a sufficiently accurate gas concentration measurement on timescales of month to years with acceptably small cross sensitivities and at a good cost-benefit ratio. Beside an initial calibration, regular re-calibrations are necessary to maintain the required measuring accuracy. The small size of NDIR sensors allows for the construction of underwater instruments that can be deployed on various carriers ranging from stationary buoys and deep-sea moorings to mobile research vessels and remotely operated or autonomous underwater vehicles (AUVs and ROVs).

Recent advances in the field of narrowband IR-radiation sources and corresponding detectors should allow for the realization of compact and specialized tunable diode laser absorption spectrometers (TDLAS) for hydro carbon molecules and $CO_2$ measurements in the near future. Advantages over NDIR detectors with respect to size, stability, cross sensitivity, signal noise, linearity of sensor response and accuracy are expected. By that autonomous underwater greenhouse gas measurements on wide temporal and spatial scales could be greatly improved.

# The use of MIR spectroscopy for localization of threat substances in urban societies

**Lars-Erik Nilsson, Pierre Strömbeck, Bengt-Göran Andersson and Ola Norberg**

*Portendo AB, Sweden*

Recent events in Europe as well as in other parts of the world have highlighted the serious problem of terrorists attacking civilians with homemade bombs. The explosives used in many of these bombs are manufactured by simple chemistry in e.g. ordinary kitchens. During the synthesis process, which might be of several weeks duration, the processes used often emit specific gases that can be detected by MIR-spectroscopy and thus reveal the illegal activity before any harm is done.

The FP7 program LOTUS (Localization Of Threat Substances in Urban Societies) aimed at developing means for efficient location of such activities. The concept relies on a fleet of vehicles equipped with fast, specific and very sensitive gas sensors that in combination with GPS for location and GSM for communication sends real time data to a central, reporting time and location of suspected gas emissions.

Several sensor techniques were tested in the project. The most viable solution was based on MIR spectroscopy comprising a tunable quantum cascade laser and a special Herriot cell with a very long path length. Sensitivities in the ppb region at data rates of a few Hz were obtained. Field trials both in urban environments and the country side in several European countries clearly demonstrated the practical possibility of real time detection of illicit synthesis of explosives over large areas.

# Selective trace gas detection through CEAS and diffusion time of flight

**Anthony E. Miller, Jason McKeever, C. Ricardo Viteri and Bruce Richman**

*Entanglement Technologies, 42 Adrian Ct., Burlingame, USA*

Entanglement Technologies will report a novel approach to optical detection of trace vapor using a combination of cavity enhanced absorption spectroscopy and diffusion time of flight. The direct optical detection of large molecule vapors presents a number of unique challenges due to low vapor pressures, a lack of resolved spectral features, and the presence of interfering species with overlapping absorption spectra. By recording the changing optical absorption as sampled atmosphere diffuses into a target-free buffer gas, the concentration of target molecules may be determined using a Bayesian statistical signal processing technique. This technique avoids the need for laser wavelength scans while simultaneously providing robust background rejection. The use of fixed laser wavelengths enables cavity-locked absorption measurements with high data acquisition rates and eliminates the need for precision wavelength monitoring. The technology is currently under development for a variety of molecules including trace hydrocarbons, benzene, BTEX, and TCE. Experimental demonstration of the simultaneous detection of multiple species of hydrocarbon molecules will be reported.

# QEPAS based sensor for detection of sulfur dioxide using a CW-DFB-QCL

**J. P. Waclawek[1], R. Lewicki[2], M. Jahjah[2], Y.F. Ma[2], E.t.H. Chrysostom[1], B. Lendl[1] and F. K. Tittel[2]**

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A sensitive, selective and compact gas sensor based on quartz-enhanced photoacoustic spectroscopy (QEPAS) employing a 7.25 µm CW-DFB-QCL was developed for detecting $SO_2$ at ppbv concentration levels, suitable for environmental and industrial monitoring.

QEPAS is a sensitive technique that allows measurements of trace gases in an ultra-small absorption detection module with a total sample volume of a few $mm^3$ [1]. A 32.768 kHz quartz tuning fork (QTF) is used as a sharply resonant transducer for acoustic waves, which are induced in an absorbing gas by modulated optical radiation. The QTF is a piezo-electric element which converts its deformation by pressure waves into separation of electrical charges that can be measured as a voltage. A QEPAS signal enhancement can be obtained by adding tubes to the sensor architecture acting as a micro-resonator. Moreover, for slow relaxing molecules, blending the gas mixture with

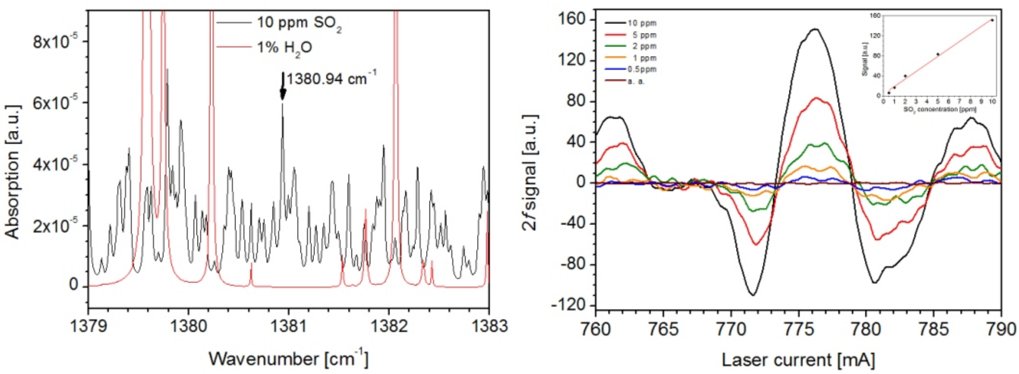


**Fig. 1** (left) HITRAN simulated absorption spectrum of 10 ppm $SO_2$ and 1% water vapor ($p$=100 torr, $l$=1 cm); (right) 2$f$ wavelength modulation QEPAS signals acquired at different $SO_2$ concentration levels (a.a.: ambient air). The inset shows the linear dependence of measured 2$f$ signals as a function of $SO_2$ concentrations.

water vapor, which is an efficient catalyst for V-T relaxation processes in the gas phase, can improve the detected QEPAS signal [2].

Using 2*f* wavelength modulation spectroscopy (WMS) the DFB-QCL emission wavelength was tuned across an $SO_2$ absorption line centered at 1380.94 $cm^{-1}$ (Fig. 1 left). The generated acoustic waves were detected by the QTF. The sensitivity and linear response of the QEPAS based $SO_2$ sensor were investigated at different $SO_2$ concentration levels (Fig. 1 right). For a certified mixture of 10 ppm $SO_2$ in $N_2$, the determined QEPAS SNR was 100, which yielded a minimum detection limit (1 ) of 100 ppbv.

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# Measuring system for hazardous substances detection

**Bartłomiej Jankiewicz[1], Marcin Miczuga[1], Krzysztof Kopczyński[1] and Robert Owczarek[2]**

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Hazardous chemicals detection apparatus based on cascade lasers have been recently widely used in advanced safety control systems aimed at counteracting terrorist attacks and in natural environment monitoring. Owing to the great progress in the cascade lasers technology and spectroscopic detection methods that has been made in the last years, it is possible to detect the concentration of hazardous molecules at the ppb (part per billion) level. This allows detecting the explosives and toxic materials storage sites, including toxic weapons, as well as monitoring these materials transportation.

In this work, a modular system dedicated to detecting the hazardous chemicals, built with the implementation of a cascade laser, is presented. Apart from the laser, the main elements of the system are: a multipath cell, an IR detector and a control and data analysis unit. The operation of the measuring system is exemplified by its application to detecting the ammonia residual molecules concentration in the air.

# A new method for absorption feature resolution from wavelength modulation spectroscopy (WMS) Signals

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One of the main issues with WMS technique applied to Tunable Diode Laser Absorption Spectroscopy (TDLAS) for gas sensing comes from the relationship between absorption features and the demodulated signals. In such experiments, the actual absorption signal is masked by the modulation process and thus sensitive to its parameters and to the physical behaviour of the components involved in the setup. Moreover, absorption parameters such as line width and shape become just artifacts at the end of the modulation-demodulation chain and are not easily detectable.

The detection of those parameters, which are at the fundamentals of any gas spectroscopy device operating on samples subject to unknown physical conditions, is now possible through a fitting technique which is aware of the modulation scheme. Another advantage of this technique arises from the fitting of experimental WMS data, improving measurement accuracy and immunity from interference and enabling the detection of weak absorption signals.

The key of this method stands in the source emission and modulation being modeled in both power and wavelength; this model is then fed to an absorption simulation to achieve a synthesized WMS signal. By optimizing the parameters in the absorption simulation through a least-square fitting routine, the experimental signal is fitted against the synthesized signal, giving the absorption parameters as an output.

This method has been tested and applied to a WMS-TDLAS system for oxygen detection in closed containers, enabling simultaneous concentration and pressure measurements.

# Sponsors

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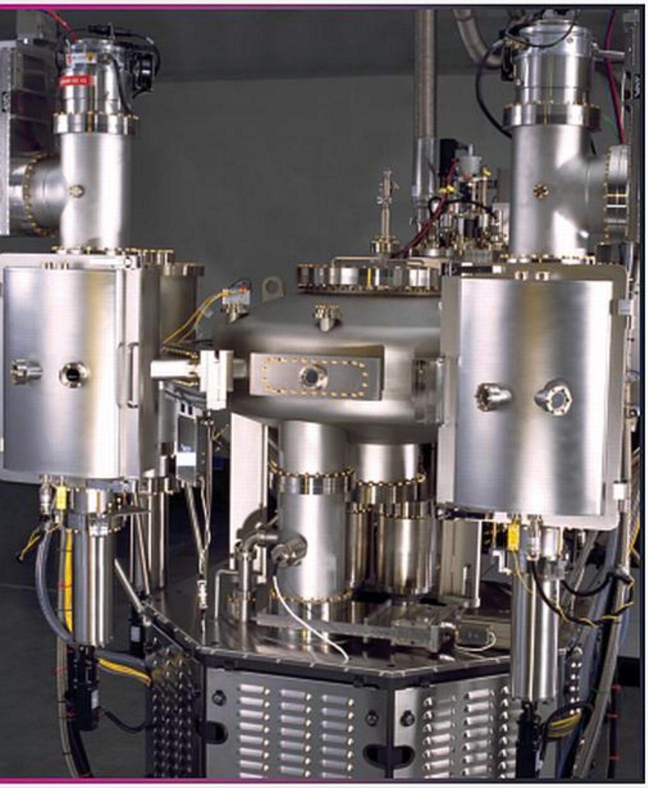

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Crystal Fibre • *aero*LASE • Koheras • SuperK

# SuperK MIR Compact
# - seeing the invisible

- **1500-4200 nm continuous spectrum**
- **Output power > 75 mW above 1.5 µm**
- **Single mode fiber delivery**
- **All colors in one beam**
- **Rugged mechanical design**
- **No moving parts**
- **Room temperature operation**
- **Compact footprint**
- **Operation at the press of a button**

NKT Photonics is the world leading provider of supercontinuum sources in the visible and near infrared range.

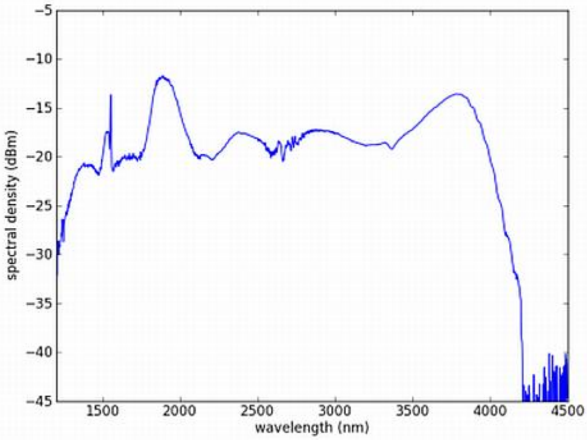
The SuperK MIR supercontinuum extends the spectrum into the mid-infrared by using zirconium fluoride fibers.

## Prelimenary

| Specifications | |
|---|---|
| Spectral Range | 1500-4200 nm |
| Power | >75 mW |
| Repetition Rate | 30 kHz |
| Master pulse width | <3 ns |
| Output fiber mode | Single mode |

## Applications

- **Source for FTIR spectrometers**
- **Remote spectroscopic analysis of atmospheric compounds**
- **Illuminators for thermal imaging**
- **Explosive detection**
- **Detection of hazardous chemicals**

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COMEF

Scientific and Research Instruments

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