# PROJECT FINAL REPORT

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# Final publishable summary report

# **Executive summary**

The WideLase project focused on compact, rugged and cost effective laser sources with a wide tuning range for safety and security applications. Novel Interband Cascade Laser (ICL) structures with wide gain bandwidth were realized enabling room temperature continuous wave operation in the mid infrared (MIR) wavelength range. Monolithic concepts for electrical tuning based on multi-section DFB as well as acousto-opto-electronic lasers were investigated for the first time in the wavelength range of interest. Based on these novel MIR sources, three particular challenging applications with significant market potential were investigated within the project. Laser based sensor systems targeting alcohol sensing for traffic control and monitoring of formaldehyde emission during wood board manufacturing as well as a laser based sensor for hydrocarbon leak detection were successfully developed in the WideLase project.

The technical work had been structured into three work packages:

- WP1: Material development for broad gain bandwidth
- WP2: Widely Tunable Laser Sources
- WP3: Laser based Sensing for Safety and Security

In order to reach the project goals, significant challenges had to be overcome in various fields, ranging from epitaxial semiconductor growth via laser design and processing to mid infrared sensor development. The WideLase consortium comprises renowned research groups, academic and industrial SME partners from across Europe with a range of complementary competencies covering all aspects from semiconductor material development to photonic components and sensor systems.

In parallel to these technical activities, also work was performed concerning the future use and exploitation of the WideLase results including Intellectual Property Rights (IPR) and market studies. In addition, project dissemination by publications and presentations was performed, which also included the maintenance of a project related website (www.widelase.eu) and the preparation of the workshop MIRSENS organized by the WideLase consortium.

# **Project context and objectives**

# Concept and main ideas of the WideLase Project

The WideLase project focused on the development of widely tunable, mid-infrared semiconductor lasers for the detection of hazardous substances. Due to their compact size and narrow linewidth, semiconductor lasers are ideal light sources for tunable laser spectroscopy (TLS). The development of Quantum Cascade Lasers (QCLs) and Interband Cascade Lasers (ICLs) has extended the wavelength range accessible with semiconductor lasers into the mid-infrared region. This spectral range is very important for chemical detection and identification, as nearly every molecule has characteristic and strong absorptions in this wavelength range, leading to high sensitivities. Up to now, almost all sensing applications utilize semiconductor lasers with a fixed (or narrowly tunable) emission wavelength, as e.g. distributed feedback (DFB) lasers. The emission wavelength of these devices is fixed by a grating structure that is incorporated into the laser structure and can only be tuned over a very limited range by temperature or current variation. Therefore, these devices are not suitable for the detection of multiple gas species or the spectroscopy of chemicals with a wide absorption range. Semiconductor lasers with a wide tuning range in the mid-IR are available, but they use external cavities which make the lasers bulky, costly and sensitive to shock and vibrations.

We investigated novel monolithic concepts for electrical tuning combined with interband cascade material to realize compact, rugged and cost effective laser sources with wide tuning range for safety and security applications in the 3.3 to 7.0  $\mu$ m wavelength region. These sources were then be utilized for three different important safety and security applications: remote detection of alcohol vapor in exhaled breath, remote monitoring of formaldehyde and hydrocarbon leak detection.

#### Application 1: Remote detection of alcohol vapor against drunk driving

For this application, a sensor was developed that remotely identifies passing vehicles in which the concentration of alcohol vapor from breath in the passenger compartment is above a certain threshold. The detection is made by laser spectroscopy where the radiation is invisible and not harmful to the eyes. This system should be able to identify and quantify concentrations of alcohol down to the legal limit in vehicles passing at high speed (target up to 100 km/h). When a car has been identified an indication is sent *e.g.* to a police patrol that stops the vehicle and the driver is asked to produce a conventional breath test for legal identification of drunk driving. The system shall be transportable, easy to set up and automated. A schematic of the scheme is shown in fig. 1.

Today the police enforce the drunk driving regulations by a two-step procedure. In the first step, a screening of potential offenders is made at sobriety check-points. Breath analysis using handheld Alco meters are used to sort out the individuals that are possibly driving beyond the legal limit for alcohol intoxication. In the second step, blood sampling or more advanced breath analyzing instruments are used to provide judicial evidence. The first sorting phase is generally very inefficient since the check points have to be positioned to avoid impeding the traffic. The procedure also involves a large amount of randomness where a large number of vehicles have to be checked. The probability to catch someone driving under the influence of alcohol by a random check is very low, around 0.5%. Using TLS, it is possible to monitor the concentration of ethanol in the exhaled breath of a person confined within the passenger compartment of a motor vehicle. Using the broad absorption feature of ethanol around  $3.3~\mu m$ , a high sensitivity and selectivity can be achieved.

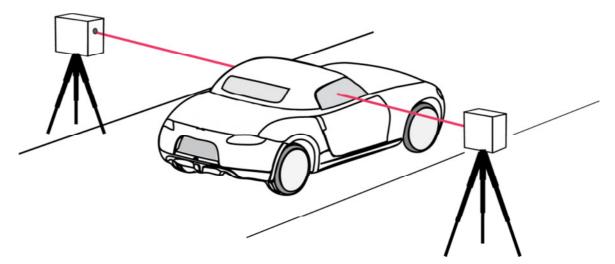


Fig. 1: Schematic view of the sensor concept for remote detection of alcohol vapor

The legal limit for driver intoxication is in some countries based on the blood level of alcohol while others have approved the use of breath analysis as evidence. A conversion factor of 2100 between the breath alcohol value and the blood alcohol content has been established and is today generally accepted. The mass of alcohol per mass of blood given in ‰ is most common for defining blood alcohol concentration. The alcohol level at which a person is considered to be legally impaired varies by country. In most of Europe the legal limit is 0.5 ‰ while in some countries such as Sweden, Norway and Poland the limit is 0.2 ‰. Using the conversion factor above, these levels corresponds to alcohol concentration levels in exhaled breath of 133 ppm and 53 ppm respectively.

#### Application 2: Remote monitoring of formaldehyde in wood-board manufacturing

Formaldehyde (H<sub>2</sub>CO) is the most important aldehyde produced commercially, and is used in the preparation of urea-formaldehyde and phenol-formaldehyde resins. It is also used in consumer products, such as shampoo, toothpaste, disinfectants, permanent press clothing, particleboard (PB), Oriented Strand Board (OSB) and medium density fibre board (MDF). In homes, the most significant sources of formaldehyde are likely to be pressed wood products made using adhesives that contain formaldehyde. Medium density fiberboard (MDF) contains a higher resin-to-wood ratio than any other UF pressed wood product and is generally recognized as being the highest formaldehyde-emitting pressed wood product. Formaldehyde has been classified as carcinogenic to humans by the International Agency for Research on Cancer (IARC) since 2004. Formaldehyde in composites is significant enough that the California Air Resources Board (CARB) issued regulations in 2009 to limit formaldehyde emissions from composite panels used in finished consumer products made from these panels. The EPA, the Consumer Products Safety Commission and the Centers for Disease Control have identified levels above 0.1 ppm, as a concern for exposure by sensitive populations. As H<sub>2</sub>CO emission standards become more stringent there is a clear need for continuous monitoring of formaldehyde emissions during manufacturing of PB and MDF. Accurate on-line monitoring will allow manufacturers to gain tighter control of emission levels, reduce rejected lots, reduce claims, and improve profitability. Ultimately, real-time formaldehyde emissions monitoring will improve indoor air quality.

We proposed to develop a continuous on-line monitoring system based on TLS technology. The system shall be able to perform real-time measurements of the H<sub>2</sub>CO concentration in order to achieve high levels of prediction of the formaldehyde level in the finished products. The objective of the work is to develop a prototype hardware and software system to accurately detect H<sub>2</sub>CO emissions by long-path monitoring. We estimate that we could achieve a sensitivity of 5 ppb over a one meter path at an integration time of 3 seconds. We can compare this value with the proposed CARB 2009 limits for formaldehyde listed below, showing the great promise of this approach for the targeted application.

- Hard- Quality Plywood (HWPW) must be less than 80 ppb
- Particle Board (PB) must be less than 180ppb
- Medium Density Fiber board (MDF) must be less than 210ppb

#### **Application 3: Hydrocarbon leak finder**

The detection of hydrocarbons (like methane, ethane, propane etc.) in inaccessible compartments is a very important and challenging safety application with no suitable solution on the market yet. A cheap and compact, but powerful "hydrocarbon leak finder" has a huge market potential with applications everywhere where hydrocarbons are guided through tubes and hydraulic fluids are in use like e.g. gas pipelines, gas stations, refineries and air planes. Also, leakage detection of aerosol cans by measuring traces of propellants has been defined as application test case within the WideLase project. Nowadays, laws dictate that each aerosol can has to undergo a "bubble" test in a hot water bath. This is very cost- and time-consuming and manufacturers are searching for new techniques that make these tests faster, cheaper, and more directly. TLS is a promising approach and the requirements on a TLS sensor for aerosol can monitoring are summarized as follows:

Primary propellant	Propane (C <sub>3</sub> H <sub>8</sub> )
Secondary propellant	i/n-Butane (C <sub>4</sub> H <sub>10</sub> )
Min. leakage rate	< 0.12 cc/min
Speed	200 – 250 bottles/min
Background gas	Ambient air
Temperature	Ambient
Pressure	Ambient

In principle for hydrocarbon leak detection, two configurations are possible: A local measurement with e.g. a photo acoustic cell or a more global measurement with a line-of-sight set-up. While in a typical safety application, the selectivity is not of high importance and a sum-concentration measurement is acceptable, an additional exploitation possibility will be the replacement of expensive gas chromatographs by cheaper, but more selective laser spectrometers based on newly developed laser sources. The broad tuning range of the devices investigated within the WideLase project is an important prerequisite to cover a wide variety of absorption lines based on the different targeted hydrocarbon species.

#### Widely tunable interband cascade laser sources

All these three sensing applications in safety and security (as also a variety of other applications) will require compact, rugged and cost effective laser sources with wide tuning range and extended output power, which are not available on the market today.

The novel laser sources as developed within the WideLase project will be based on innovative interband cascade laser (ICL) material with broad gain bandwidth. Interband cascade lasers combine the interband transitions on which diode lasers are based with the cascade scheme as also utilized in quantum cascade lasers. This hybrid laser has proven very well suitable to cover the mid-infrared spectral range where nowadays room temperature continuous wave performance can be obtained in the entire 3 to  $\sim$ 6  $\mu$ m range. This wavelength range is not completely accessible either with diode lasers or quantum cascade lasers if room temperature continuous wave performance is required. In this regard, ICLs are the ideal choice to address laser challenges in this project, by developing cost-effectively widely tunable gain material covering the 3.3 to 7.0  $\mu$ m spectral range based on one material platform.

ICLs exploit a special band alignment available in the antimony material system for the cascading scheme. The laser design utilizes optical transitions between the conduction and valence bands in a staircase of GaSb-based type-II QWs (see fig. 2), while tunnelling of electrons out of the valance band into the conduction band of the injector/collector region (named InAs/Al(In)Sb multilayers) generate holes in the valance band by the leaky quantum well concept which enables interband transitions (indicated by an arrow). As for quantum cascade lasers, the emission wavelength of ICLs can entirely be designed by the thickness of the constituting quantum well layers, while the emission wavelength of ICL shows, however, a simple linear dependence on the QW thickness.

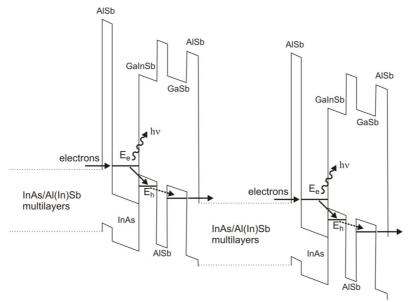


Fig. 2: Band structure of GaSb based type II interband cascade active region.

Compared to quantum cascade lasers which are at the moment the only serious competitor to cover the entire wavelength range of interest in this project, ICLs have the main advantage that they operate at more than an order of magnitude lower power while delivering ample of power for the targeted spectroscopic applications. Design and epitaxy of such lasers will be carried outin the project, supported by advanced material characterization performed. In addition to the extension of the wavelength range, the available gain bandwidth and single mode tunable emission wavelength range has to be significantly extended.

The ICL approach used in WideLase will bring important benefits for the realization of compact, rugged and cost effective laser sources with wide tuning range and extended output power as targeted in the present proposal. Since ICLs can operate at more than an order of magnitude lower power compared to QCLs, this then allows the effective development of widely tunable devices requiring multi segments to be contacted (which also avoid the necessity to mount the devices epi-side down, as usually required in case of QCLs).

Two different approaches will be used within the WideLase project for the realization of widely tunable lasers based on ICL material with broad gain bandwidth: multi-section lasers with binary superimposed gratings (BSGs) and widely tunable acousto-opto-electronic lasers.

#### Multi-section lasers with binary superimposed gratings (BSGs)

Most commonly used in the field of gas detection are conventional distributed feedback DFB lasers (including also QCL DFBs). These devices can be monolithically fabricated as single semiconductor chip and can be highly miniaturized. Due to the lack of moving parts they are extremely rugged and maintenance-free devices compared to external cavity lasers with macroscopic grating. However, their tuning range is intrinsically limited. To overcome this obstacle and preserve the advantages of monolithic lasers, one approach investigated within the WideLase project is based on multi-section lasers with binary superimposed gratings (BSGs). The device concept will offer a rapid switching of the emission wavelengths to a plurality of discrete spectral positions within spectral gain region of the laser medium as required by the applications within WideLase. The devices will allow to addressing multiple spectral positions each of which are tunable around their respective center wavelength. Based on IC active material with an extended gain bandwidth, target WideLase device performance figures include a total tuning range of 200 nm.

#### Widely tunable acusto-opto-electronic lasers

The second approach investigated within the WideLase project will be based on the novel and patented device technology: The approach here is based on a multi-component laser design with no moving parts in which the wavelength selective component is an acoustic wave. This design allows to introduce feedback and

modulation of the emitted laser light by electronic control of the properties of the acoustical wave. The resulting laser will have the unique advantage of intrinsic simplicity, fast tunability and low power consumption (in the order of 500-700mW). The technology also does not need multiple etched gratings nor complex phase or temperature control.

However, in order to realize precise control of the opto-acoustic interaction, a further monolithic integration is necessary. This will be supported by detailed multi-physics simulations. This monolithic integration into a compact widely tunable laser will be performed within the WideLase project for the first time. The acousto-opto-electronic laser approach is considered more risky than the concept based on multi-section lasers with BSGs as described above, but has a great inherent potential for further commercial exploitation in various fields of application (not only in sensing but also telecommunications). Based on ICL material, final target performance figures of the targeted acousto-opto-electronic laser source within the WideLase project includes a continuous tuning range of 200nm and a tuning speed of less than 10 milliseconds.

### Main S&T results/foregrounds

In general, ICL devices with state-of-the art performance figures have been obtained within the consortium in the targeted mid-infrared (MIR) range. Figure 3 shows the threshold power density of diode lasers, various interband cascade lasers (ICLs) and quantum cascade lasers (QCLs) as a function of emission wavelength. The threshold power density of diode lasers (black squares) increases rapidly for wavelengths larger than 3  $\mu$ m. ICLs (blue and green squares) on the other hand have an almost constant threshold power density in the wavelength range between 3 and 6  $\mu$ m, with particularly low values in the 3 to 4  $\mu$ m range. The threshold power density of QCLs (red squares) is more than one order of magnitude larger. This demonstrates that the ICL approach is most competitive in the wavelength range of interest.

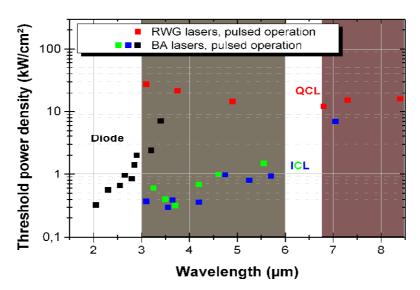


Fig. 3 Threshold power density for various devices concepts in the MIR wavelength range between 2 and 8.5  $\mu$ m.

Based on the new ICL material from WP1, widely tunable devices were developed in WP2 and corresponding devices were supplied to the WP3 application partners for the development of highly sensitive detection systems in the following fields:

- Laser based sensor for remote detection of alcohol against drunk driving,
- Laser based sensor for formaldehyde monitoring,
- Laser based sensor for hydrocarbon leak detection.

Focus with respect to laser device development in the last period of the project especially concentrated on the realization of specific performance parameters in the target wavelength range of interest as defined in close interaction with the application partners for device delivery to WP3, making best use of the available technology base.

Based on the developed laser devices, sensor systems have been successfully developed in all of the above application fields as will be discussed in more detail in the corresponding WP3 section of this report. Field test have also been performed for all developed systems in the last period of the project.

As an example, the sensor systems of the WideLase project for remote monitoring of alcohol is shown in figure 4 below.



Fig. 4 The laser based sensor system of WideLase for remote detection of alcohol against drunk driving.

In the following, the objectives of the various WideLase workpackages are reviewed:

#### Development of high quality ICL material

The objective of **WP1** was the development of high performance interband cascade laser material, which was optimized with respect to operation temperature, power consumption, emission wavelength and gain bandwidth. The work performed in WP1 included the design of interband cascade layer structures, the epitaxy of laser layers, and the characterization of ICL material and devices. Two different material systems were investigated in the WideLase project: **ICLs based on GaSb substrates** for the wavelength range from 3 to 5.5  $\mu$ m and **InAs based ICLs** for longer wavelengths from 5.5 to 7  $\mu$ m.

High performance **ICLs based on GaSb** have become the main workhorse material system of the project. Based on several design optimizations, state-of-the-art ICL structures with respect to threshold power density, temperature stability and wavelength reproducibility were grown within the entire  $3-5~\mu m$  wavelength range. Typical threshold current densities of lasers in this  $3-5~\mu m$  range are below 300 A/cm² at room temperature, which allows continuous wave (CW) operation of the devices. A record value below  $100~\text{A/cm}^2$  was also demonstrated. In addition, the work on **InAs based ICLs** for the longer wavelength range beyond  $5.5~\mu m$  was also progressing further and it was possible to demonstrate CW operation of a DFB device in the  $6.5-7~\mu m$  wavelength range.

The careful optimization of the active region and the waveguide design of GaSb based ICLs has lead to a substantial reduction of the threshold current densities, allowing CW operation at room temperature in the spectral range from 3 to  $5.2 \, \mu m$ . As shown in figure 5, the threshold current densities are below 200 A/cm² in the 3 - 4  $\mu m$  window (with a record value as low as 98 A/cm²), and below 300 A/cm² for the entire 3 - 5  $\mu m$ 

range. The threshold current densities of the less mature InAs ICLs are around 1 kA/cm $^2$  in the 5 - 7  $\mu$ m region. Data from competing groups (GaSb ICL – NRL Washington, InAs – University of Oklahoma) is also included in figure 5, showing unique availability of state-of-the-art laser material in both material systems in the WideLase consortium.

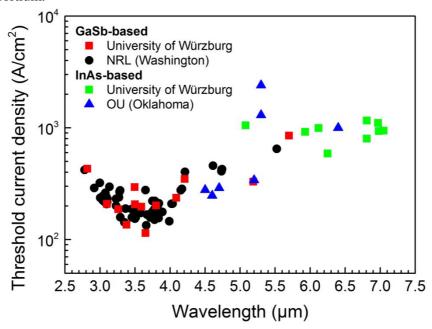


Fig. 5 Threshold current densities for GaSb and InAs based ICLs in the  $3-7 \mu m$  emission wavelength range

Figure 6 (a) shows the temperature dependent threshold current density  $j_{th}$  of ICL based three broad area lasers emitting at 3, 4 and 5  $\mu$ m respectively in pulsed operation (1 kHz, 250 ns).  $T_0$  values between 42 K and 61 K have been achieved, which represent a state-of-the-art temperature stability. Figure 6 (b) show the corresponding spectra at room temperature. Processed as narrow ridge device several lasers operated in cw mode up to temperatures of 80 °C.

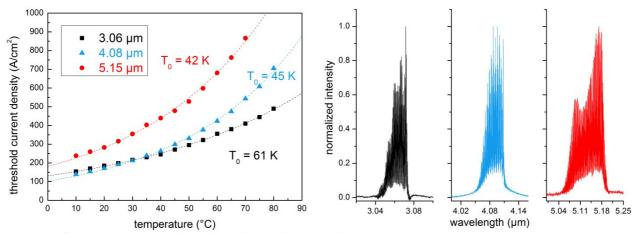
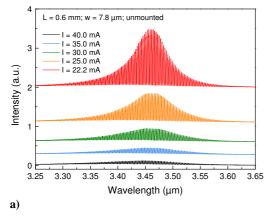
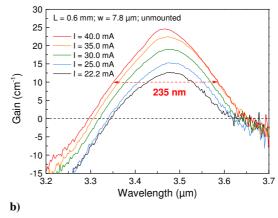


Fig. 6 a) Temperature dependent threshold current density for three broad area ICLs (2mm x 150 μm). b) Spectra for three ICLs in different wavelength regions.

In order to determine the gain bandwidth up to the laser threshold, amplified spontaneous emission (ASE) spectra were measured from different lasers, followed by an extraction of the gain curve based on the Hakki-Paoli method. Since the spectral resolution of the FTIR spectrometer is limited to 0.12 cm<sup>-1</sup>, short lasers (0.6 mm cavity length) had to be used to increase the longitudinal mode spacing. The Hakki-Paoli technique is only applicable up the threshold of the lasers, therefore an AR coating was applied on one facet of the devices. While the continuous wave threshold current of an uncoated 0.6 mm long laser was 22.2 mA, it almost

doubled after the facet coating and ASE spectra could be measured up to I = 40 mA as shown in figure 7 (a). Figure 7 (b) shows the calculated gain curves for various injection currents between 22.2 mA and 40 mA.





*Fig.* 7 a) ASE spectra of a 0.6 mm long ICL driven with different currents. b) Gain curves of the same laser driven with different currents. Assuming an overall resonator loss of 10 cm<sup>-1</sup>, a gain bandwidth of more than 200 nm is achievable.

As expected the increased carrier density results in a wider gain and higher peak gain. Having in mind the increase of carrier density above threshold, a gain bandwidth for ICLs can be estimated for the corresponding wavelength region. Assuming a threshold gain of 10 cm<sup>-1</sup> (accounting for 7.6 cm<sup>-1</sup> mirror loss for a 1.2 mm long laser with uncoated facets and some additional loss from the chromium grating in distributed feedback lasers), a gain bandwidth of 235 nm at an injection current of 40 mA is demonstrated in the 3.5 µm region.

#### Widely tunable Lasers

The objective of **WP 2** was the development of widely tunable laser sources based on the ICL material provided by WP1. Two different approaches are used within the WideLase project for the realization of widely tunable lasers based on ICL material with broad gain bandwidth: multi-section lasers with binary superimposed gratings (BSGs) and widely tunable acousto-opto-electronic lasers based on surface acoustic waves (SAW).

DFB type devices have also been used for covering more than one channel and probing the gain bandwidth of the developed ICL structures for widely tunable device processing and subsequent optimization. An example of these gain bandwidth studies is given in figure 8 showing spectra of two DFB laser devices: To evaluate the gain bandwidth of the material at 3.3 µm, DFB laser devices were processed with the corresponding DFB grating periods varied over a wide range *on the same ICL EPI wafer material*. The devices show emission around 3270 nm and 3475 nm, respectively. The observed DFB emission wavelengths are 205 nm apart, thus confirming the required gain bandwidth of the underlying material for widely tunable devices.

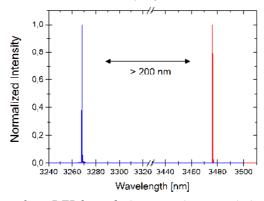


Fig. 8 Spectra of two DFB laser devices covering an emission wavelength range exceeding 200 nm based on identical ICL material.

#### ICL based widely tunable multi-section lasers with BSGs

The schematic design of a multi-section laser and corresponding micrographs from device processing are shown in figure 9.

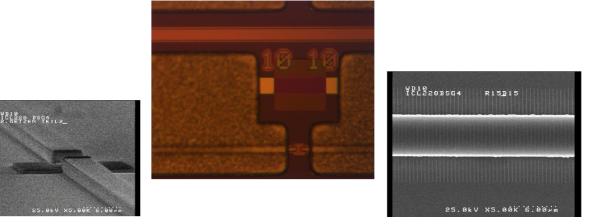


Fig. 9 Optical and SEM micrographs from widely tunable BSG device processing concerning the definition of the BSG grating structure and the multi-section device structure. A special challenge in device processing concerns the realization of a suitable electrical separation between the sections of the laser in order to eliminate electrical crosstalk.

Theoretical device simulations were used to evaluate suitable BSG structures based on the gain characteristics of the WP1 material and various widely tunable devices have been fabricated and characterized. Special emphasis in the reporting period concentrated on the realization of specific performance parameters defined in close interaction with the application partners for device delivery towards WP3 making best use of the BSG technology base. This particularly relates to specific wavelength channels needed for the application and the tuning range coverage within these channels.

Figure 10 shows an example the wavelength tuning behaviour (mode map) of fabricated widely tunable devices. These ICL BSG devices have been delivered to the WP3 application partners for subsequent sensor development and related testing. The devices were prepared to cover specific absorption features of the respective application.

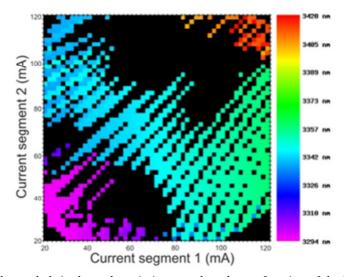


Fig. 10 Mode map (color-coded single mode emission wavelength as a function of the injected currents in both segments) for a widely tunable multi-section laser with BSGs prepared to cover specific alcohol gas absorption features in combination with allowing a corresponding baseline measurement at lower wavelength.

The mode map was recorded at a fixed laser chip temperature.

An example of a BSG ICL device covering a total tuning range around 200 nm in three discrete channels is shown in Figure 11.

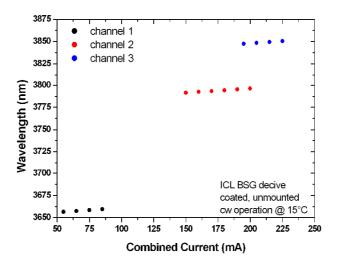


Fig. 11 A widely tunable ICL device in the 3.8 μm range with three discrete channels covering a wavelength range around 200 nm. The device was operated at a fixed laser chip temperature.

Widely tunable acousto-opto-electronic lasers

With respect to the development of an acousto-optically tunable laser (see figure 12), the content of the performed work was centred around:

- interdigitated transducer process engineering and testing,
- monolithic integration of piezoelectric "booster layer",
- full device integration and testing.

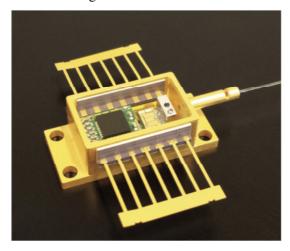


Figure 12 Illustration of an acousto-optically tunable laser including RF-generator chip (green), laser and fibre alignment module within an industry standard butterfly package.

Interdigitated transducers (IDTs) receive the RF signal and transform it into a surface acoustic wave by means of actuating the underlying piezo electric layer. The resulting SAW will permeate the optical field of the laser and induce a feedback grating. The IDTs consist of hundreds of metal transducer pairs with a minimum feature size and spacing of 100 nm as shown in figure 13.

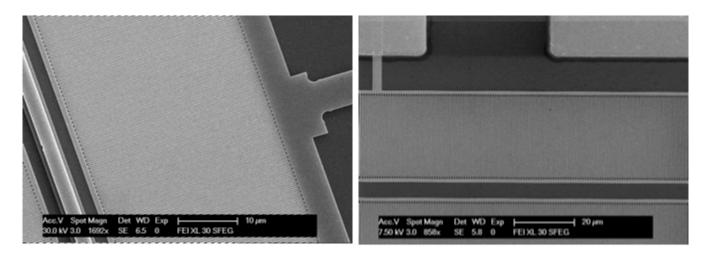
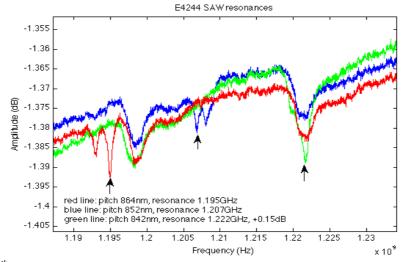


Figure 13 SEM images of high quality IDT transducer structures.

Summarizing the desired tuning effect of the acousto-optically tunable laser by means of on-chip generation of acoustic waves could not be realized within the time frame of WIDELASE. The main obstacles that were encountered include unforeseen major design changes and process additions and as a consequence process control challenges. However, promising results on monolithic piezo-integration with laser material have been demonstrated. Figure 14 shows RF-resonance test results based on a set of fabricated integrated laser devices with AlN booster layer. SAW resonances are visible however weak. The different resonances correspond to different IDT pitches and were designed to cover a wider range of the gain bandwidth of the laser material.



**Figure 14** 7<sup>th</sup> order SAW resonances of an acousto-optically tunable test structure laser PHOENIX lasers.

The three lines correspond to three different IDT pitches.

#### **Sensor Systems**

The objective of **WP 3** was to use the novel widely tunable laser sources developed within WP 2 for the realization of innovative sensor systems. For all targeted fields of applications of WideLase (alcohol against drunk driving, hydrocarbon leak detection as well as formaldehyde monitoring) novel laser-based sensor systems have been developed and corresponding field test have been performed in the last period of the project.

# Sensor system for remote monitoring of formaldehyde

A sensor system for remote monitoring of formaldehyde during wood-board manufacturing has been completed as shown in figure 15.





Fig. 15 The fully operational formaldehyde sensor.

The retro-reflector that defines the measurement path is shown in the background

The formaldehyde sensor runs all control and measurement software autonomously. For displaying and data logging the sensor communicates over Ethernet to a PC or a Lap Top. The software enables further processing of the measurement values like averaging and logging. The software also sets parameters for operating the sensor. The sensor system sensitivity has a performance exceeding the target.

Sensor specification summary;

- Real time sensing the platform response time is below 0,5 second
- High selectivity application of TDL technology and the algorithms ensures no interference from other constituents in the measurement path
- High sensitivity the HCHO detection limit for the field instrument is below 40 ppb per meter
- In-situ monitoring the analyzer platform provides reliable measurements of formaldehyde vapor in large spaces (e.g. wood board production halls)
- EPA compliant the GasEye HCHO measurements are provided with sensitivities required by the EPA regulations
- Maintenance free GasEye HCHO can be equipped with a self calibrating feature, minimizing the maintenance costs
- Robustness the platform provides reliable measurements in harsh environment. GasEye HCHO is insensitive to dust, smoke, temperature variations and optics contamination

The formaldehyde sensor has also been successfully tested in a field trial at a furniture manufacturing plant as shown in figure 16. The objective of the study was to test the formaldehyde emission levels at different manufacturing stages from the board cutting station, through the milling station and to the final treatment.

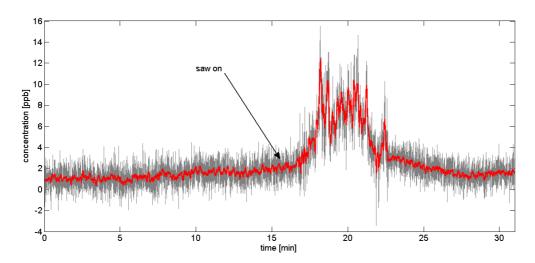


Fig. 16 Variation in formaldehyde concentration during sawing of an MDF board

### Sensor system for remote monitoring of alcohol

A sensor system for remote monitoring of alcohol has been successfully developed. The system has been used in several field trials and has obtained widespread publicity in newspapers and television. We give a summary of the main results below.

The system is based entirely on the GasEye platform in a retro-reflective configuration. The remote alcohol detection system was first tested in the lab using a miniature car cabin with a length of 75 cm with integrated Volvo 740 side windows. An artificial breath sample corresponding to 106 ppm EtOH vapour concentration, equivalent to 0.05% BAC (the legal limit in most EU countries), was introduced into the cabin. In figure 17 we can see that without EtOH in the sample the system detection limit (2 $\sigma$ ) reaches 0.36 ppm in the 75 cm long cabin at integration time of 0.25 seconds. This corresponds to 0.27 ppm\*m *i.e.* the detection limit of the system exceeds the targeted value by roughly a factor of 3. When the 106 ppm sample was flowed through the cabin we could see the expected increase of EtOH concentration recorded by the GasEye EtOH system. After the sample flow is stopped and eventually the cabin ventilated we observe the detected EtOH to go back to zero level again.

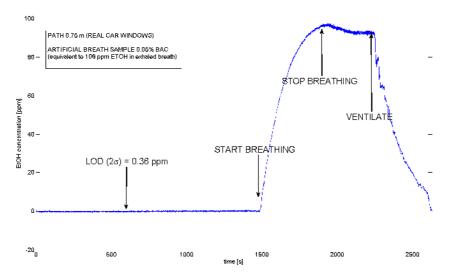


Fig. 17 A log of the alcohol concentration as a function of time. The alcohol is released after 1500 seconds in the plot. We see that the lower detection limit  $(2\sigma)$  is 0.36 ppm

As a next step we setup a system for validation in real settings. For this purpose we have used a Nissan SUV, see figure 18. The preliminary results are similar to the lab experiment using the miniature car compartment.



Fig. 4.20 Validation test utilizing a Nissan SUV. 106 ppm of alcohol vapour is generated in the passenger compartment utilizing a Breath Test Simulator

Figure 20 shows a scene from a television show in the German channel N24 where we demonstrated the ability of the system to detect alcohol in the passenger compartment from a moving vehicle.

PROMILLETEST 04.04.2015

Laser erkennt betrunkene Autofahrer aus der Entfernung



Fig. 20 Detecting alcohol in the passenger compartment of a moving vehicle using the laser based alcohol sensor

#### Hydrocarbon leakage detector for aerosol cans

Leakage detection from aerosol cans by measuring traces of propellants has been defined as additional application within the WideLase project. Nowadays, laws dictate that each aerosol can has to undergo a "bubble" test in a 50 °C hot water bath. This is very cost- and time-consuming and manufacturers are searching for new techniques that make these tests faster, cheaper, and more directly.

First, a test jig has been designed to be able to simulate the application in the lab. Measurement results (see figure 21) using the developed laboratory system demonstrate the targeted performance for the monitoring of aerosol cans on a conveyor belt

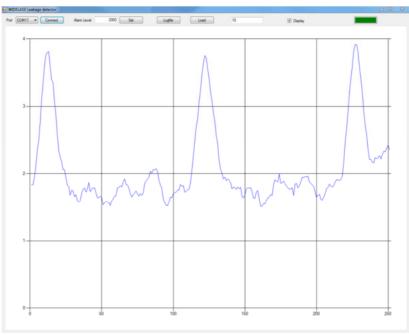


Fig. 21 Measurement based on the developed laboratory system with a leakage rate of 0.12 cc/min.

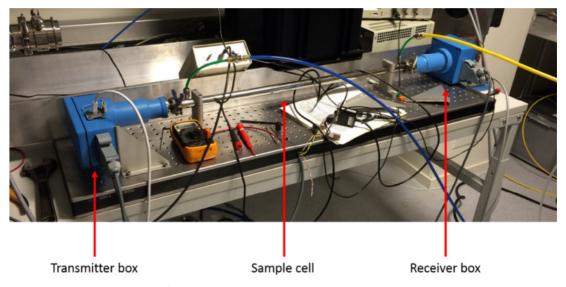


Fig. 22 Leakage detector mounted for testing.

Finally, the laboratory system was converted into a system for field testing (Figure 22) and corresponding measurements were successfully performed together with an application partner: Leakage rates smaller than 0.12 cc/min were measured.

# Potential impact, exploitation of results and main dissemination activities

#### **Innovation related impact**

The overall objective of this project is the realization of a new generation of widely tunable laser sources in the mid infrared wavelength range  $3.3-7~\mu m$  and photonic sensors using laser spectroscopy based on these novel sources. Currently laser based sensing applications are limited by the properties of the available light sources, which do not allow to address the wide tuning range required in the wavelength range of interest for many sensing application of organic substances. Since the devices developed within WideLase will constitute compact, rugged and cost effective laser sources (compared to e.g. external cavity laser ECL based sources) various additional fields of application will benefit from this new development.

The impact of the project in future markets depends on significant technological innovations in material and device design for increased tunability, device processing and sensor system development. For example, the maximum tuning range of application grade lasers (cw operation at RT) are significantly extended by using novel approaches in ICL material and device fabrication. Using this material and innovative device designs widely tunable laser sources were developed for the first time in the wavelength range of interest. For monomode devices overgrowth free technologies are used, which have the potential to increase the device yield and thereby to reduce the device cost compared to overgrowth based approaches considerably. This is especially important for the GaSb system, for which overgrowth is difficult.

The availability of these MIR laser sources in conjunction with innovative signal-processing schemes lead to significant improvements in the sensing performance compared to currently established technology. Laser based sensing systems with previously unavailable sensitivity and functionality therefore become available. The planned work was driven by the impact such new sensors will have in important market segments in the detection and monitoring of hazardous organic substances.

Within this project devices with extended tuning range were developed. This is of great interest especially also for gas sensing of multicomponent gas mixtures in general. Moreover widely tunable devices are required for many additional applications (also including medical applications), especially if the analysis of liquids is required, where in general broad absorption features are prevailing.

# **Societal Impact**

A major driver for the realization of monomode laser sources is the development of a new qualitative level of monitoring techniques using photonic sensors especially for the detection and monitoring of hazardous organic substances. By elaborating innovative sensing methods, this will facilitate the introduction of new applications for environmental gas sensing, industrial process control or also medical applications.

Within the proposal, technical results are already directly validated in important safety applications. The availability of improved sensing techniques will therefore directly contribute to the well being and health of the population by e.g. monitoring toxic gas constituents (investigated within WideLase: "Remote monitoring of formaldehyde during wood-board manufacturing") or leakage hazards (investigated within WideLase: "Hydrocarbon leak finder").

Also the remote detection of drunk driving was investigated within WideLase. Although alcohol-related traffic accidents have been decreasing throughout the European Union, driving whilst under the influence of alcohol continues to be an important cause of road traffic crashes.

The active involvement of four industrial beneficiaries ensures that the project results will be fully exploited commercially. This will clearly have a positive direct impact on job creation.

#### **Economical impact**

The market for gas sensors is of global significance and is rapidly expanding, fueled by new applications based on ever-growing public concerns over such issues as pollution, health and safety at work, and developments in industrial process control. Particularly in the area of infra-red gas sensing there has been a significant growth in recent years.

An area with particular potential to increase this market still further is Tunable Diode Laser Spectroscopy (TLS), which is now seeing commercial exploitation. A variety of gas species can be detected at trace levels in the ppm or even ppb range using TLS. Applications range from the industrial (e.g., combustion diagnostics, leak detection), environmental monitoring, to medical applications (e.g. optical breath analyzers or blood glucose level sensors). The key components of modern TLS systems are semiconductor lasers with narrow line width, tunability over one or more gas absorption line and high side mode suppression ratios to ensure high spectroscopic selectivity. Such lasers also allow the whole system to be miniaturized and allow quantitative measurements without the need for recalibration, thus offering the advantages of ease-of-transport and lower cost. The market for Gas Sensing and Analysis based on traditional techniques has an annual growth around 3-4%. TLS and other laser based instruments on the other hand are increasing their sale by more than 20% annually. This growth is not at the expense of the traditional techniques but largely coming from new applications opened up by this technique. Once technologically mature, the sensors developed within this project will therefore strongly contribute to new applications.

At present, mainly optical and chemical methods are used for the detection of trace gases. Chemical processes are often not selective enough and only possess a limited sensitivity. Optical techniques using absorption measurements currently use thermal light sources as well as lasers. The greatly increased intensity of lasers for a certain spectral range compared to thermal emitters allows the sensitivity to be increased by many orders of magnitude. In addition using monomode lasers a very specific target wavelength can be chosen preventing any overlap in absorption with other gas components, allowing an extremely selective measurement to be achieved. Monomode lasers are therefore key components in compact state-of-the art TLS set-ups.

Present DFB lasers, however, possess an inherently limited wavelength tuning range and application-grade, compact, rugged and cost effective widely tunable lasers are not available in the wavelength range of interest. The project WideLase is overcoming these obstacles hindering a widespread exploitation of laser based sensing for many applications where an extended tuning range is required as the ones addressed within WideLase. There are also excellent chances to open new markets in the area Process Analytical Chemistry if robust widely tunable light sources finally become available.

The European level was essential for obtaining access to the range and quality of personnel, technical expertise and resources required to undertake the present research. Only within a European approach was it possible to establish such a focused vertically-integrated research project, which allowed to cover all the required expertise within a cost-efficient small, yet highly competent consortium. WideLase also contributed to creating useful and durable industrial links between European industries and academia.

#### **Exploitation of results**

The primary exploitation of the project results will be the future commercialization of the devices developed and investigated within the project. In this respect the project is very well positioned. The active involvement of four industrial beneficiaries provides several routes to commercial exploitation in the period after the conclusion of the project. In addition, the academic beneficiaries will contribute to the exploitation e.g. by the education of engineers and scientists in the program or the publication of research results in scientific papers and at conferences.

The beneficiaries of the Project WideLase form a consortium where each of the members has developed outstanding skills in its own working area. Special care has been spent already when selecting the consortium to realize efficient synergies between beneficiaries. The industrial beneficiaries of the project are focused leading companies in Europe covering all required steps for laser based sensor fabrication.

The results obtained within the WideLase project will also be directly used in further research activities and projects.

Concerning education, the knowledge acquired during the project will be promptly made available in seminars and lectures at the academic beneficiaries. It will especially be used for training courses carried out by post-graduate students. Highly qualified Ph.D. students were educated within the project, giving students first hand insight e.g. into innovative mid infrared device growth, fabrication and characterization, strengthening their position in the job market and thereby also further improving the technological competitiveness of companies with positive impact for European economy.

Non-commercial exploitation also occurred by the beneficiaries including the publication of research results in scientific and wide audience journals as well as presentations at conferences or the education of students in areas related to the project. This increased the awareness on advances in laser based sensing techniques in the broad public, which in turn results in additional market opportunities.

Overall the project beneficiaries are in a very strong position to fully exploit the results commercially and non-commercially in a wide variety of areas in environmental and industrial gas sensing applications with additional potential for medical and fluid sensing applications.

#### Main dissemination activities

The dissemination of the knowledge and specific results formed an important part of the project and was covered by its own work package.

Dissemination especially covered the following activities:

- Maintain and continuously update the project dedicated website
- Disseminate the project activity to a broader public by press releases etc.
- Publish papers in scientific journals on the technical project achievements
- Report the technical project achievements at international conferences, but also to use international conferences for providing a general overview of the project activities and of the benefits of the novel photonic components developed within WideLase to a scientific audience and to potential industrial users
- Organize an international workshop on *Opportunities and Challenges in Mid-infrared Laser-based Gas Sensing*.

#### Website

The project website was created at the very beginning of the project duration at <a href="www.widelase.eu">www.widelase.eu</a>. A screenshot of the home page is shown below.



The website is divided into two main parts: public and internal. The information of its different sections has been continuously updated as the project proceeded. The internal has been secured and password protected with the restricted access for the consortium partners only. It contained mainly various confidential documents to be circulated between the partners of for their direct access at any time of the project duration: templates of documents, files of the reports and deliverables, the presentations from the progress and review meetings, collection of patents in fields of interest for the consortium, publications and conference abstracts prior the submission, etc. In the public part, beside the information on the Project itself and on the consortium and its particular partners, there are successively placed the current news (e.g. on the organized workshop or important events as WideLase on TV programme) and all various forms of the project results and other outcomes: text of the press release, list of published papers with a direct link to full text at the publishers' website, info on the media events with the involvement of the consortium, or on the publications in popular magazines related to the project or its achievements.

#### **Press Releases**

At the start and the end of the project **press releases** have been prepared.

#### Novel European Sensing Systems for a Greener and Safer World

In the recently launched project WideLase a new generation of laser sources for sensing will be developed by a joint effort of European scientists and engineers. A major driver for the realization of these laser sources is the development of a qualitatively new level of techniques for the detection and monitoring of hazardous organic substances.

The availability of improved sensing techniques will result directly in benefits for society and the well-being and health of the population. Within the project technical findings will be instantly validated by two industrial partners of the consortium in important safety applications: A real-time online monitoring instrument for formaldehyde will be investigated by Airoptic (Poland) within the project which also has great commercial potential with emission standards being enforced on products that emit formaldehyde particularly including plywood products. A second application pursued by Airoptic concerns the remote detection of drunk driving. Although alcohol-related traffic accidents have been decreasing throughout the European Union in recent years, driving whilst under the influence of alcohol continues to be an important cause of road fatalities. In addition, a hydrocarbon leak finder will be investigated by Norsk Elektro Optikk (Norway) within WideLase preventing fire hazards as well as preventing impacts on world climate and global warming by reducing methane emissions in sectors encompassing oil and gas production or long distance gas transmission.

cmissions in sectors encompassing oil and gas production or long distance gas transmission. Those are only examples of the ways in which the newly developed laser sources can bring about benefits to the public. The underlying laser technology will facilitate a diversity of other applications which will have a positive impact on safety and security while simultaneously contributing to a greener world and having an enormous economic potential. WideLase will create durable links between European industries and academia and will contribute to maintain European competitiveness at an industrial level, by bringing some of the major European component manufacturing centres abead of competition in important emerging markets.

ahead of competition in important emerging markets.

Consortium partners nanoplus (Germany) and Mach8 Lasers (the Netherlands) are responsible for mono mode laser development within the project und will investigate two highly innovative concepts for wide tunability and unprecedented performance in the MIR wavelength range between 3.3 µm to 7.0 µm. nanoplus is currently an international leading supplier of lasers for gas sensing applications and notably provided devices for the current NASA Curiosity mission on Mars. Present laser sources, however, made commercially available by consortium partner nanoplus at a variety of wavelengths possess an inherently limited wavelength tuning range. Application-grade, compact, rugged and cost effective widely tunable lasers are not available in the wavelength range of interest, nanoplus therefore sees unique opportunities in the WideLase project overcoming these obstacles hindering a widespread exploitation of laser based sensing for many applications where an extended tuning range is required. The success of the four industrial partners in the various fields will only be possible by a tremendous support from the academic WideLase partners at University of Würzburg (Germany) and Wrocław University of Technology (Poland) laying the project foundation by design, growth and characterization of novel semiconductor structures for mono mode laser fabrication.

Recently, a Grant Agreement was signed between the European Union and the project coordinator nanoplus Nanosystems and Technologies GmbH. The WideLase project with partners from Germany, Norway, Poland and the Netherlands has a duration of three years and is supported by funding in the amount of Euro 2.2 million from the EU as part of the 7th Framework Programme. Establishing WideLase and a European level is essential for obtaining access to the range and quality of personnel, technical expertise and resources required to tackle the various research challenges of the project.

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The above shows the project press release prepared at the start of WideLase. Also other articles have then appeared based on the original press release (e.g. in *Laser Focus World* or *Gases&Instrumentation*):



#### WideLase dissemination in LaserFocusWorld



WideLase dissemination in Gases&Instrumentation

The project press release prepared at the end of WideLase is shown below.

Within the European project WideLase, scientists and engineers from across Europe joined forces to

Within the European project WideLase, scientists and engineers from across Europe joined forces to develop a new generation of laser based sensor systems with direct benefits for society and the well-being and health of our population.

Three challenging applications addressing important markets have successfully been realized in the project: One application in traffic control comprises a system able to remotely detect driver intoxication. Driving under the influence of alcohol continues to be a major cause of road fatalities. The new sensor developed by Airoptic (Poland) deploys an eye safe laser beam transmitted across the road, through the passenger compartment of a driving car. Laser based spectroscopy is used to measure the concentration of alcohol vapor in exhaled breath. The second application by Airoptic is a formaldehyde detection system for on-line monitoring during wood board manufacturers, Formaldehyde is a known carcinogen and the most significant sources of formaldehyde in homes are likely to be pressed wood products. Accurate on-line monitoring will allow manufacturers to gain tighter control of emission levels. The third application investigated by Norsk Elektro Optikk (Norway) is the detection of leakage from acrosol cans. By law each aerosol can has to undergo a final leakage test before it can be delivered to customers. Presently the most commonly used method is to (Norway) is the detection of leakage from acrosol cans. By law each acrosol can has to undergo a final leakage test before it can be delivered to customers. Presently the most commonly used method is to dip the bottles into a hot water bath and check for bubbles. This method is very inconvenient and time-consuming. An elegant solution based on a contactless passing-by optical measurement as desired by industrial users has been developed in WideLase.

The WideLase project (www.widelase.eu) with partners from Germany, Norway, Poland and the Netherlands started in 2012 and was supported by the European Commission with a funding of 2.2 million Euros within the EU 7th Framework programme. The European level was essential to obtain access to the range and quality of personnel, technical expertise and resources required to tackle the various research challenges of the project.

Consortium partner nanoplus (Germany), an international leading supplier of lasers for gas sensing applications, and Mackè Lasers (the Netherlands) were responsible for the development of novel laser sources within the project. They investigated two highly innovative concepts for application-grade,

sources within the project. They investigated two highly innovative concepts for application-grade, compact, rugged and cost effective widely tunable lasers in the mid infrared, which were then used for

compact, rugged and cost effective widely tunable lasers in the mid intrared, which were treen used for the development of the above sensor systems.

The success of the project's four industrial partners in their various fields was only made possible by a tremendous support from the academic WideLase partners at the University of Würzburg (Germany) and Wrocław University of Technology (Poland) laying the project foundation by design, growth and characterization of novel semiconductor laser structures.

WideLase has created durable links between European industries and academia and will contribute to maintain European competitiveness at an industrial level, bringing Europe ahead of competition in immortant engering markets.

important emerging markets.

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Press release made public at the end of the WideLase project.

#### WideLase Workshop

The WideLase consortium organized a workshop on the subject of the project in mid-infrared laser based gas sensing and related issues. MIRSENS III  $-3^{rd}$  International Workshop on Opportunities and Challenges in Mid-infrared Laser-based Gas Sensing, was held in Würzburg, Germany, 5-7 March 2015. All the information about the workshop, including its final Programme, Book of Abstracts, list of invited speakers, or a Gallery of photos, are available at the workshop website which is still active (also reachable via WideLase homepage): www.mirsens.com - see a screenshot below.



In order to disseminate the event and broadly distribute the information, a related article plus an announcement has also been published in "Gas & Instrumentation" magazine (see the figure below). G&I became also an official media partner of the Workshop. In short, the Workshop can be summarized as follows:

- about 80 participants from 13 countries,
- (Austria, Belgium, Canada, Finland, France, Germany, India, Italy, Norway, Poland, Switzerland, UK, USA),
- 2.5 day programme, 36 presentations (11 invited talks plus 25 contributed talks),
- 6 exhibitors/sponsors,
- 8 presentations from the WideLase consortium.



Pages from "Gas & Instrumentation Magazine" advertising the Mirsens 3 Workshop

#### **Publications in journals**

The consortium has been publishing the obtained results in regular scientific journals. Within three years of the project duration, there were published 20 papers, plus 3 have been submitted recently and are under review process, and a few further ones containing the WideLase results are under preparation yet, and will be published shortly after the project end. The articles have been appearing in such high rank international journals as *Electronics Letters*, *Applied Physics Letters*, *Photonics Technology Letters*, *Optical Materials Express* or *Journal of Applied Physics*, plus a topical review in *Journal of Physics D* – all these underline the importance of the achievements within the society in the international scale. The full list of these publications (including the submitted ones) is given in Table A1.

#### **Conference presentations**

The consortium disseminated very actively the information on the project and its results at international conferences and workshops in the field of sensing and photonics. International conferences (like Photonics West) also served as a platform to provide a general overview of the project activities and the benefits of the novel WideLase components to a scientific audience and to potential industrial end users.

Altogether, the WideLase consortium has given at least 70 presentations within the three years of the project duration, 19 of which were the invited talks, which underlines the importance of the achieved results. The full list of the given presentations is given in Table A2.

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