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Miniaturised photoacoustic gas sensor based on patented interferometric readout and novel photonic integration technologies (MINIGAS)

MINIGAS

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MINIGAS

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1 Executive summary

High-sensitivity gas sensors are used to measure the presence of a variety of trace gases in the environment. The gas sensors have a wide range of applications, such as, monitoring poisonous gases and air pollution from power plants, traffic, etc. The goal of the European FP7 MINIGAS project was to build a miniature gas sensor achieving two or three orders of magnitude better sensitivity than other methods with a similar size could achieve. The sensor is targeted for the low-cost market (down to 100€ per sensor unit), still having high sensitivity and selectivity for a wide range of gases. The reduction of the cost compared to the existing systems (cost >10k€) will pave the way for more widespread use of gas sensors. There are a number of potential applications for the technology, for example in health care, homeland security, work safety, and in monitoring indoor air quality and greenhouse gas emissions (such as CO_2 and methane).

Production of highly sensitive and portable gas sensors is made possible by a photoacoustic sensing technology with innovative parts. Gasera Ltd has recently developed a micromechanical cantilever-based device for detecting signals in photoacoustic spectroscopy. The mechanical movement of the cantilever, caused by the photoacoustic pressure waves, is much larger than the movement of a conventional membrane microphone, and this movement is detected using a novel interferometer. The technology enables fast response time and miniaturization, in contrast to other optical gas detection methods where long optical path lengths are needed for high sensitivity. It also enables robustness for handheld outdoor applications. The foundation of the gas sensor miniaturization, volume manufacturing and significant cost reduction is a multidisciplinary combination of novel electronics manufacturing technologies, including silicon microsystems (MEMS) and multilayer ceramics (LTCC) manufacturing technologies as well as novel mid-infrared LED and laser diode components as light sources.

Gasera is already using the component technologies developed in MINIGAS in its sensor products, and in collaboration with the end-user companies new potential products are foreseen and further investigated in the rapidly growing gas sensor markets. Moreover, the mid-infrared LED component developer and manufacturer IoffeLED is already supplying its novel devices for gas sensors.

During the final project phase, two different kind of photoacoustic sensor prototypes were implemented based on the component technologies developed during the earlier phases: A handheld sensor prototype for methane gas concentration measurements, and a simultaneous carbon dioxide and water vapour sensor prototype for atmospheric greenhouse gas flux measurements. The evaluation and testing demonstrated that the photoacoustic gas cell can be miniaturized without compromising the sensitivity.

The MINIGAS consortium was composed of research organisations and companies representing the complete value chain from technology developers and component providers to end users, namely VTT Technical Research Centre of Finland (as coordinator), Doble Transinor, Drägerwerk, Gasera, Ioffe Physical-Technical Institute, Selex Sistemi Integrati, and University of Turku.

2 Project context and objectives

MINIGAS (*Miniaturised photoacoustic gas sensor based on patented interferometric readout and novel photonic integration technologies*) was a research project under European Community's 7th Framework Programme (FP7). The project was carried out during years 2008 – 2012 and was coordinated by VTT Technical Research Centre of Finland. The other partners were Doble Transinor, Drägerwerk, Gasera, Ioffe Physical-Technical Institute, Selex Sistemi Integrati, and University of Turku. In the early stage of the project, QinetiQ Ltd also participated. The total budget for the project was EUR 2.7 million, including EC funding of EUR 1.85 million.

2.1 Context

High-sensitivity gas sensors measure the presence of trace gases. The sensors have a wide range of applications. MINIGAS project was dealing with the development of miniaturised gas sensors based on the novel photoacoustic detection technology.

2.1.1 Towards handheld optical gas sensors

The market for gas sensors is competitive, fragmented and crowded. There is a wide range of industrial, environmental and safety applications in which sensitive trace gas detection is needed, such as:

- Indoor Air Quality
- Emission monitoring
- Industrial gas detection
- Process control
- Maintenance
- Safety (combustible, toxic)
- Homeland security
- Health diagnostic
- Sports performance diagnostic

Market surveys estimate the overall global gas sensor market is >$500M/year with sensor systems worth >$3000M, and further continuous growth is expected driven by legislative requirements and emerging economies. Major findings from the gas sensor market analyses are:

- Demand is driven by the need for handheld and portable devices.
- Growth potential for environmental monitoring is large due to changing legislation.
- Increasing terrorist threat highlights need for devices with enhanced selectivity.
- Advanced technology is needed for extended calibration intervals minimizing the system maintenance cost.

There is a large variety of gas sensing methods. Optical methods are often superior to other methods because of inherent advantages in the areas of specificity, reliability, ruggedness against ambient factors, lifetime, speed of response, "non-invasiveness" and independence from material supplies.

There are both non-spectroscopic and spectroscopic methods used for these purposes. The most sensitive optical methods are based on fourier-transform infrared spectrometers (FTIR) or tuneable-diode lasers (TDLAS). Both of these methods measure optical absorbance. In long-path absorption spectroscopy, the beam penetrates through the sample gas and the absorption at two or more wavelength bands is compared. The sensitivity depends mainly on the absorption path length, radiation power and the response of the detector while the selectivity is limited by the resolution of the spectrum analyser. Because the detection limits scale linearly with the available optical path length, short path lengths (i.e. small gas cells) lead to bad sensitivity. Miniaturization of these methods is therefore not feasible. The loss in sensitivity would be dramatic. In other words, this creates great challenges when aiming for a portable instrument.

2.1.2 Photoacoustic gas sensor

Photoacoustic spectroscopy (PAS) is based on the absorption of light leading to the local warming of the absorbing volume element. The subsequent expansion of the volume element generates a pressure wave proportional to the absorbed energy, which can be detected via a pressure detector. The theoretical limitations of this technology are far from what has been achieved with any technology today. The full potential has not been reached due to the use of conventional microphones in sensing the pressure waves.

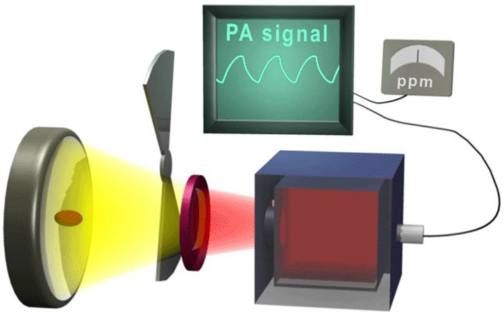

Figure 1. Basic principle of conventional photoacoustic gas sensing.

When MINIGAS was starting, Finnish SME Gasera had very recently developed a novel differential photoacoustic (PA) measurement technology. The essential part of it is a MEMS-based mechanism for detecting the pressure waves created in a photoacoustic cell, see Figure 2. This innovation can be used to realize infrared gas sensors of extreme sensitivity. Instead of the usual microphone (i.e. membrane with capacitive readout), the Gasera’s cell contains a “free-standing” silicon cantilever. The differential PA cell operates as a differential detector for infrared (IR) absorption signals propagating through the measurement and reference paths. The mechanical movement of the free end of the cantilever is 100x larger than the centre of a fixed membrane. Furthermore, the movement of the cantilever is detected interferometrically not capacitively, that is the pressure difference modulation between the chambers is monitored by probing the cantilever movement with an optical interferometer. This concept allows open-path and flow-through detection of gases.

In the differential PA sensor, it is possible to combine the advantages of long-path absorption spectroscopy and photoacoustic spectroscopy. This is because the differential PA cell equipped with an ultrasensitive optical microphone is used as a selective infrared detector similarly as in the long-path absorption spectroscopy. The gas inside the differential cell acts like an optical filter (so-called gas correlation method) providing good selectivity without a need for optical filters or spectrograph.

Furthermore, it was foreseen that the technology allows a miniaturized, but still very sensitive, gas sensor if combining the silicon cantilever microphone, high-efficiency IR LEDs as light sources, and high-sensitivity interferometric read-out system. Theoretical predictions indicated that cantilever based PA cell can be miniaturized with sensitivity up to three orders of magnitude over the prior art.

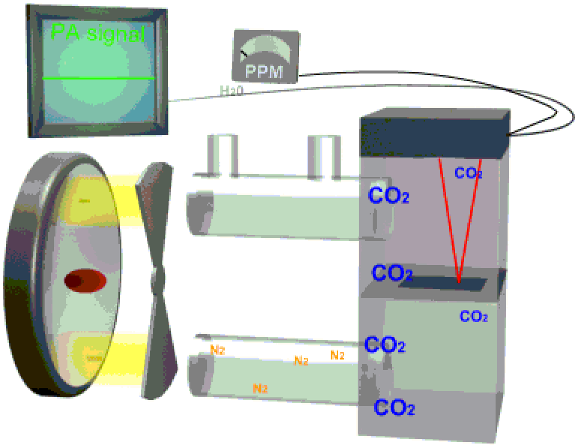

Figure 2. Principle of photoacoustic gas sensing based on the differential cell and cantilever arrangement with CO_2 as an example “target” gas.

2.2 Project objectives

The overall goal of the MINIGAS project was to design, fabricate and demonstrate a miniaturized, self-contained, integrated gas detection subsystem with the following features:

- Use of the photoacoustic measurement principle in detection.
- Use of a novel cantilever and interferometric readout method.
- Use of novel mid-infrared light emitting diodes (LED) as sources.
- Achieving specificity of response and “re-programmability” for different types of target gases, by filling a target gas into a dedicated chamber at the time of manufacturing. Thus allowing “gas sample/refresh” time of less than 30 seconds enabling the ability to be able to follow gas concentration in real time, with shorter than 1 minute warm-up time
- Sensitivity should be two or three orders of magnitude better than with other optical measurement methods having the same form factor.
- A sensor module with external electrical connections and gas connections to input and output the sample gas; therefore, providing vibration and shock resistance to a level that enables portable applications.
- Volume production costs of a sensor with LED source less than 100€ per unit (Cost of Goods).
- Overall size of the core of the sensor not larger than 5 cm^3.
- Linear dynamic range of at least 10000 times the detection limit.
- Detection limit circa 1 ppm (parts per million) for the trace gas within 1 second measurement time (for methane gas) and assuming 1 x RMS (root mean square) noise level.
- Lower than 5 W power consumption that will enable future use in battery-powered applications.
- Versatile sensor with a modular structure allowing applicability to a wide range of gases including: CH_4, CO_2, CO, NH_3, …
- Applications including: leak detection, safety, homeland security, air quality

The project was to fabricate and test prototype miniaturized gas sensors and benchmark them against existing sensors in the following applications, as represented by the participants with end-user expertise: Homeland security (Selex), natural gas leakage detection CH_4 (Dräger), Greenhouse gases, such as, CO_2 and SF_6 (Doble).

Figure 3 shows the target positioning of the MINIGAS sensor within the gas sensing development roadmap. MINIGAS sensor is between an existing photoacoustic sensor product by Gasera, and an example of a micro-sensor system integrated on a package with the footprint of a few millimetres.

Figure 3. left) Gasera PA101 Photoacoustic detector; middle) Schematic 3D model of the MINIGAS sensor, size $5cm^3$; right) Micro-sensor system integrated on a package (note: the photo does not represent a gas sensor).

3 S&T results and foregrounds

Implementation of a super-sensitive, portable measuring device is made possible by a photoacoustic gas sensor with innovative parts combined and developed in the project, such as, a miniaturised interferometer and a miniaturised PA cell including a MEMS cantilever. While a laser is needed as infrared light source for the highest sensitivity, novel IR LEDs developed in the project are sufficient for measuring fairly small gas concentrations in lower cost portable devices.

During the MINIGAS project, two different kinds of photoacoustic sensor prototypes were implemented based on the developed component technologies: A handheld sensor prototype for methane gas concentration measurements, and a simultaneous carbon dioxide and a water vapour sensor prototype for atmospheric greenhouse gas flux measurements.

3.1 Sensor concept – Path to miniaturization

A benefit of the cantilever enhanced differential photoacoustic sensor is that the detection limit is independent on the size of the gas cell, thus giving potential for miniaturization.

The following technology portfolio was available within the project consortium and enabled the development of the miniaturised PA sensor:

- Physics and modelling of PA and IR spectroscopy
- Mid-Infrared LED sources with customized wavelengths
- Silicon MEMS Cantilever pressure sensor
- Novel type of Spatial Interferometer for cantilever readout
    - Note: In the beginning of the project, implementation of readout interferometer was also investigated using hollow waveguide (HWG) technology based on MEMS processes, but that work was discontinued when QinetiQ left the consortium.
- Multilayer ceramic circuit technology (LTCC) for manufacturing of miniaturized PA cell
- 3D integration of optics, mechanics, (opto)electronics, gas cells, MEMS, LEDs
- On-board drive, detection and readout electronics
- Signal processing for selectivity and sensitivity
- End-user requirements

These technologies and their development are described in more detail in the following.

3.1.1 Sensor concept

The initial concept for MINIGAS PA sensor presented in Figure 4. The infrared light sources provide the optical power for the gas concentration measurement based on absorption and the photoacoustic effect. In MINIGAS, an infrared LED was used as the primary infrared light source. The output of the IR source is coupled to the measurement (sample) and reference gas cells. The measurement cell is filled with a sample of the air (atmosphere) to be assessed. Whereas, the reference cell is filled with a neutral gas, such as nitrogen, which does not absorb in the specific IR LED emission band. The IR radiation from the reference and measurement cells passes into the differential photoacoustic cell. The PA cell has two absorption chambers with a common wall in which the silicon cantilever is mounted. The two chambers of the photoacoustic cell are filled (during assembly of the sensor) with the gas whose concentration in the measurement cell is to be assessed. When the light from the IR source passing through the measurement cell it is attenuated due to the presence of an absorbing gas, the light intensities in the two chambers of the photoacoustic cell become unequal. In turn, because of the photoacoustic effect, the pressures in the two chambers become different which results in displacement of the cantilever tip. The displacement of the cantilever tip is then accurately measured by the optical read-out interferometer. After calibrating the complete gas sensor system accordingly, the magnitude of the displacement of the cantilever provides information on the presence and concentration of the gas species, which the sensor was designed to detect.

In the actual gas measurements, the fundamental signal information has to be supported with information about operating environment and the final measurement result is processed using the available versatile information in an advanced information processing system. It is also noteworthy that in some cases the reference gas cell can be omitted, and, instead of splitting the output of the IR source (LED) into the two paths, it is possible to use two separate IR sources (LEDs) assuming their output powers are properly balanced.

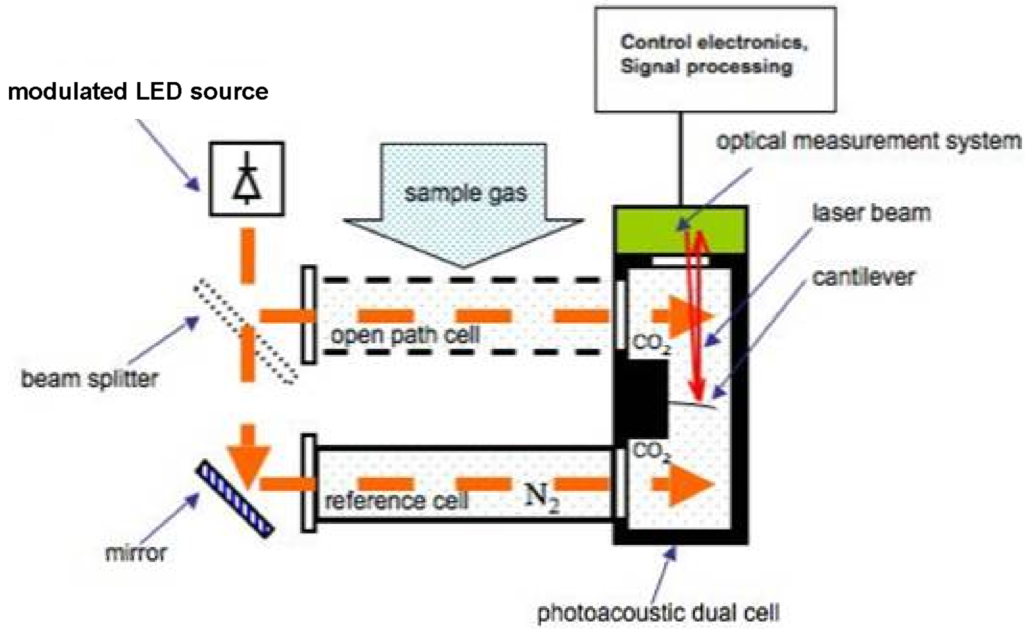

Figure 4. Initial concept of differential cantilever PA sensor with CO_2 as an example "target" gas in the PA cell.

3.1.2 Approach

The sensor is a complex multi-technological system, which makes the cost and performance optimization a challenging task. The project was using modern design and simulation tools to identify the most critical sub-systems of the sensor. The manufacturing and assembly requirements and finally the optimal structures of the sub-systems were achieved by simulations and by the implementation and evaluation of prototypes.

The chosen modular approach helped in tailoring the Photoacoustic Sensor Engine for the applications of the end-user partners. The main target applications were gases, which can be measured with the LED sources provided by Ioffe. In addition, laser diode sources were investigated for higher sensitivity and for wavelengths at which LEDs are not feasible yet. The initial modelling was done for CO_2 gas, but in the later work the primary target gas was methane. The predicted performances of the sensor concept helped the end-users to select the appropriate applications. Throughout the project, the in the technical requirements and target application were monitored by the industrial partners and by the feedback from the dissemination activities. In addition, the performances and manufacturing costs of the sensor were estimated.

3.2 Development of components and sensor subsystems

MINIGAS gas sensor consists of five important sub-systems. These are as follows:

- Photoacoustic gas cell with integrated MEMS cantilever subsystem
- IR light source (LED or laser) subsystem coupled to the measurement and reference gas cells
- Read-out interferometer subsystem
- Hybrid integration system for optomechanical integration of the three aforementioned subsystems
- On-board electronic sensing, drive and readout circuit subsystem

The subsystems and the key components in them were studied and developed to meet the overall sensor specifications as well as to enable implementation of the sensors demonstrators.

3.2.1 Silicon MEMS cantilever pressure sensor

The cantilever microphone for monitoring the pressure changed between the cavities of the photoacoustic cell was implemented by the use of silicon micromachining (MEMS) technology. The “free-standing” cantilever tip is moving due to photoacoustic pressure waves between the two gas cavities. Below picometer displacements of the tip can be detected with the optical readout, i.e. with high-precision interferometric measurement.

The MEMS cantilever needs to be mounted in the wall between the two chambers of the differential absorption cell using appropriate sealing techniques. As the movement of the cantilever is to be measured using the read-out interferometer operating with red (650 nm) laser source, the optical reflection properties of the cantilever and its optical alignment with respect to the interferometer must be taken into consideration. These factors affect the manufacture of the cantilever and the alignment accuracy when mounted within the PA cell.

The cantilever was designed for compensation of external vibrations. When planning this project, it was originally thought that it may be necessary to design a sensor containing two cantilevers, one exposed to the target gas and the other one only exposed to the mechanical vibrations experienced by the package. However, another technical possibility to combat “vibration noise” also exists. The frequency range of the mechanical response of the cantilever can – through suitable design and miniaturization – could possibly be pushed to such high frequencies (approx. >100 Hz) that the 1/f characteristic of the typical vibrational background noise no longer interferes with the measurement. This concept was analysed and proven by modelling in detail.

The MEMS cantilever was implemented as shown in Figure 5. The fabrication process was based on silicon-on-insulator (SOI) and dry etching technologies. The cantilever was etched from the front using reactive ion etching (RIE), and then from the back using deep RIE to define the cantilever within a membrane. The buried oxide was then removed from the cantilever to realise a freestanding structure. The cantilever chips were manufactured by VTT. Air gaps were made using ICP RIE. Au layer was deposited and patterned on the top of the cantilever and frame in order to produce high reflectivity for readout interferometer probing. In addition, metal contacts were sputtered on the backside of the chip to enable solder mounting onto the photoacoustic cell.

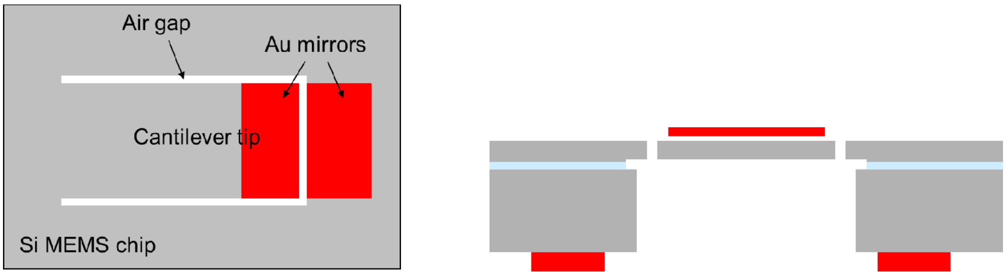

Figure 5. Structure of the cantilever chip fabricated in SOI MEMS process (silicon in grey, metal coatings in red, buried silicon oxide layer in blue, not to scale); left) top view; right) cross sectional view.

3.2.2 Differential photoacoustic cell miniaturised using LTCC technology

The photoacoustic cell developed is a differential absorption cell. In conjunction with the MEMS cantilever, it forms the functional heart of the miniature gas sensor. The MEMS cantilever is mounted in the dividing wall between the two chambers of the differential cell. The pressure difference produced between the two chambers due to the photoacoustic effect results in movement of the cantilever with the resulting displacement being measured by the read-out interferometer. The differential absorption cell also requires gas feed ports to allow pump-out and gas fill procedures during the manufacturing of the sensor, and it requires highly transmitting windows to allow incident radiation from both the infrared source and the visible light interferometer to enter the cell.

A miniaturized differential gas cell is needed to obtain PA sensor miniaturization targets. Obviously, the differential gas cell including selective sample gas has to be hermetic in order to maintain the proper gas content inside the chamber even in harsh environmental conditions, i.e. to meet an appropriate low-leak-rate specification. The key asset for targeted volume processibility of the sensor with reduced cost is the use of high-volume 3D integration and manufacturing technologies already matured in electronics industry. It is not possible to implement such a single piece miniature hermetic photoacoustic cell structure based on traditional CNC tooling by using alternative ceramic materials or metals. And gas cell volumes would be too large for fabrication by silicon micromachining. However, the hermetic differential gas cell with required geometry can be fabricated by use of the Low Temperature Co-fired Ceramics (LTCC) substrate technology.

LTCC is a mature technology used to implement multilayer electronic circuits in 3D ceramic substrates. LTCC technology benefits are good manufacturing tolerances and mass producibility, that is LTCC-based micromodules are high-volume produced and enable chip-on-board assembly at the panel level. The technology enables a novel implementation of a hermetic differential photoacoustic cell for gas sensing due to the intrinsic material properties and capability to implement precision cavities to form the 3D differential cell structure as well as hermetic sealing between IR window materials, such as sapphire, and the substrate material. The close match of the LTCC material coefficient of thermal expansion to the silicon cantilever and interferometric readout system reduces the thermo-mechanical stresses induced in the packaging. It is also possible to implement the laser driving and detector control and preamp electronics on the LTCC, if needed. In addition, the cell cavities can be coated with metal pastes to increase the reflectivity of the cavity surface in order to improve the S/N ratio in the measurement. Because of the aforementioned properties, LTCC was chosen as a fabrication technology for the differential PA cell.

Reflective metal coating of the cell walls is preferred because LTCC dielectric absorbs light. Silver and gold pastes by thick-film coating technique were tested and applied onto the cell walls. Reflectance measurements of processed samples showed that reflectivity of the substrate material can be improved by a factor of 15 to 90 in the 3...8 µm spectral band.

The implemented differential PA gas cell structure included two 8 mm cylindrical cells, diameter 2.4 mm, for reference and measurement chambers coated with a silver paste. Pictures of the differential LTCC cell structure and of the fabricated cells are shown Figure 6. The chambers were drilled at the laminated stage before the co-firing. Reflective metal coatings were deposited by the thick-film method. To assemble a complete LTCC-based cell, first the MEMS cantilever was mounted into the cell, then the windows were mounted and sealed, copper tubes were soldered to the gas inlet and outlet openings of the cell, and finally the cell was filled with the target gas through the tubes and the tubes were sealed. The dimensions of the completed photoacoustic cells were 2.5 x 2.5 x 10.0 mm.

The hermeticity of processing solder joints between copper tubes and LTCC substrate were previously tested in other projects. In this case the gas inlet and outlets were used to fill the differential PA cell with 3.5 grade methane gas. The 3.5 grade means that the gas contains less than 0.55 ‰ other gases than methane. The methane flow was guided to the inlet from gas bottle in order to cause slight overpressure to the cell, while the outlet was kept open. The methane was allowed to flow through the cell about 30 minutes before the inlet copper tube was flattened by the flattening tool. The outlet tube was flattened immediately after that. The flattened tubes were cut and the end of tubes were sealed by the soldering technique,

A sapphire window was hermetically sealed on top of the differential gas cell structure in order to probe the displacement of the silicon cantilever tip inside the sealed differential cell. Two other sapphire windows were hermetically sealed on the differential cell to enable coupling of the infrared signals into the cell. The sealed methane gas in differential cell produces selectivity against other possible gases at the measurement path. All sapphire windows were sealed with solder glass paste designed for low temperature hermetic sealing of ceramic, glass, semiconductor and metal parts. Fine leak of the sealed modules was tested by a He-leak test. The measured He-leak rate was $<2.0\times10^{-9}$ atm×cm^3/s, which fulfills the requirements for the leak rate according MIL-STD 883.

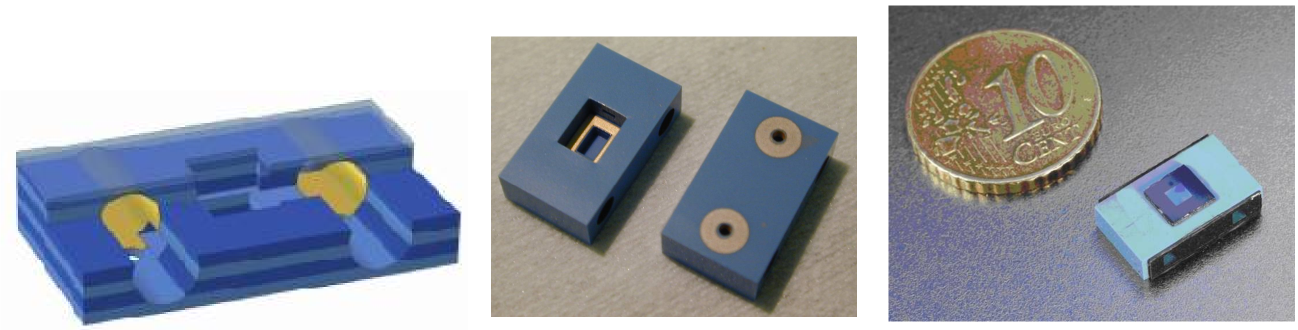

Figure 6. Differential PA cell based on LTCC carrier technology; left) structural design of the LTCC body; middle) photos of the LTCC body from the top side with cantilever mounting area and from the bottom side with gas inlet & outlet openings; right) photo from top side of the PA cell after hermetic sealing with windows for cantilever readout and for IR measurement and reference signals.

3.2.3 Spatial readout interferometer

The aim of the read-out interferometer studies was to develop a miniaturized optical interferometer capable of measuring cantilever displacements of the order of 1 pm and to be suitable for integration into the MINIGAS sensor, i.e. having dimensions compatible with the overall sensor volume. The main targets for the interferometer performance were as follows:

- Resolution of the interferometer well below 1 pm with 1 s measurement time

- The maximum allowed relative error in the displacement measurement is 1%, but the performance will be superior if the error is equal or less than 0.1%.

The novel spatial interferometer concept based on creation of interference by wave-front splitting was selected. The laser beams reflecting from the cantilever tip and the cantilever frame create an interference pattern, which can be detected with an image sensor. By further signal processing, very tiny displacements of the tip can be monitored from the interference signal.
The spatial interferometer has been developed by Gasera and was selected for the MINIGAS sensor after the optical waveguide based interferometer investigated in the beginning had appeared unsuitable for the project.
The spatial interferometer has two remarkable advantages:

- Expensive beam splitting optics is avoided by creating interference by wave-front splitting
- High temperature stability is achieved by using the cantilever frame as the reference mirror

First, design and tolerance analysis for spatial interferometer was performed and a large-scale prototype of the spatial interferometer was implemented. According to the performed tests to the spatial interferometer, the sensitivity of the interferometer was under 1 pm, which enables high sensitivity in gas concentration measurements.
Later in the project, a miniature-scale spatial interferometer prototype was designed, implemented and tested for the MINIGAS sensor demonstrator. The final designed interferometer consisted of a focused beam 670-nm VCSEL laser diode, an angle mirror which splits laser beam in two, and a CMOS linear image sensor, as depicted in Figure 7.

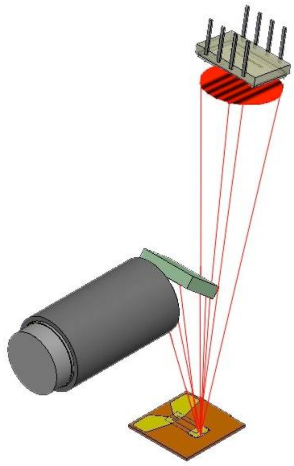

Figure 7. Spatial interferometer system prototype design layout.

3.2.4 Mid-infrared light emitting diodes

Infrared LED sources, as compared to conventional IR emitters, enable lower consumption and the possibility for high modulation frequency by current pulsing with frequencies up to several MHz. Since the output power of LEDs is constant up the several MHz, the modulation frequency in practice can be selected to optimise the total response of the sensor. The reasonably narrow emission band of the LED makes it possible to build selective detectors without any optical filters. Furthermore, the small size of the LED relates to enhancement of the optical coupling with the PA cell. These features, together with the small size and power consumption of LEDs make them advantageous for sensor miniaturization, i.e. for implementation of portable and precise instruments. LED sources are less expensive than laser diodes, which are also under development and emerging into mid-IR wavelengths and require precise current and temperature controllers.

The development of IR-LED sources within the MINIGAS project was based on the Ioffe Institute research results on narrow-gap semiconductor structures, such as, InAs and InAs-based structures, which are capable of emitting mid-IR radiation (3…7 µm). These semiconductor heterostructure devices cover broad spectral range with many important gas bands within it, as illustrated in Figure 8 where gas absorption spectra and compared with the LED emission (or photodiode responsivity) spectra. The graph on right shows methane absorption spectra measured by the use of an InAs IR-LED. According to the graph, the LED spectrum fits nicely with the gas spectrum. When filling the differential PA cell with methane, the spectral lines of methane represent the lines at which the differential PA cell is sensitive to absorbed radiation; therefore, the spectral lines corresponding methane absorption are selectively monitored by the gas sensor based on the use of the differential PA cell.

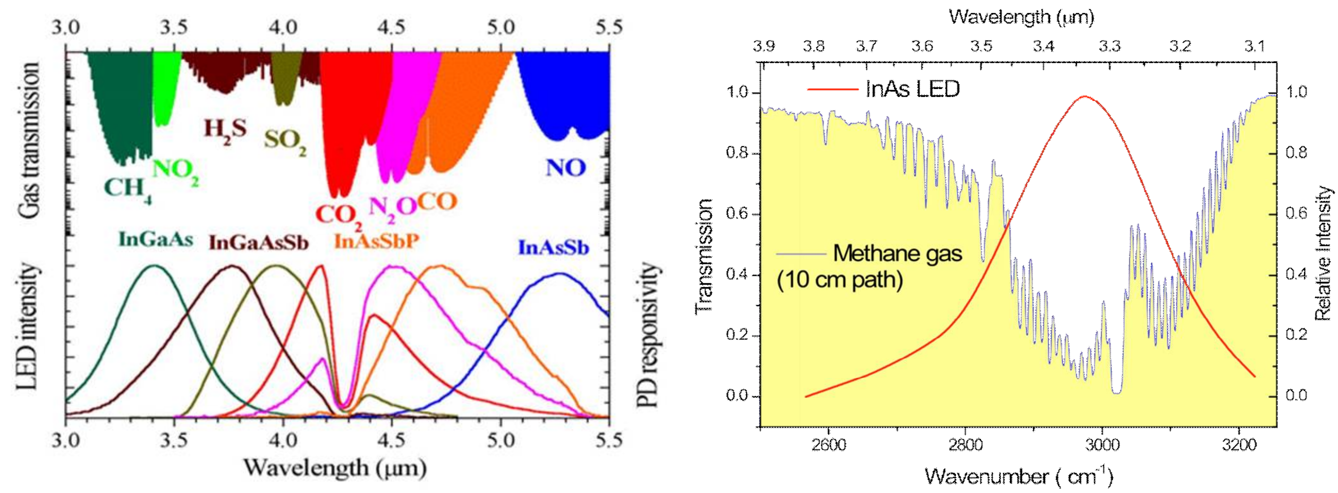

Figure 8. Transmission spectra of various gases versus emission spectra of various InAs-based IR-LEDs (on left), and transmission spectrum of methane and emission spectrum of InAs LED in more detail (on right).

Both electrically pumped and optically pumped IR-LED structures were investigated and prototypes devices were fabricated in the project. New features of double heterostructure devices used for LEDs emitting at 3.3 – 3.4 µm - plateaus in C-V characteristics and bias voltage increase - were experimentally observed and analyzed. The developed heterostructure chips enable flip-chip assembling technology and mounting of an immersion lens on top of the broadband infrared output surface for enchanced out-coupling. In addition, technique for fabrication mid-IR LEDs, including LED arrays, with 2D photonic crystal (PC) structure patterned onto the emitting surface was developed. The PC structure was investigated in order to increase the out-coupling from the chip and thus the overall efficiency. The output of PC IR-LEDs exhibits periodic structure in the near- and far-field electroluminescence measurements together with the increase of the efficient emitting area. An example of studied LED architecture is presented in Figure 9. It provides good performance and compactness thanks to: (a) good heat dissipation due to minimal distance between p-n junction/active area and heatsink and (b) efficient radiation out-coupling enabled by the broad mirror anode, deep mesa etching (leading to reflection from the mesa side walls shown as dashed arrows), and reduced total reflection losses by interaction of the photons with the 2D photonic crystal on the out-coupling surface. InAs and InAsSb based diode structures were processed into flip-chip devices with typical active areas size of 300 µm and with reflective contacts by the use of a multistage wet photolithography method.

LED devices were tested and the detection limit for several gases in photoacoustic measurements with a cantilever microphone were estimated. LED arrays exhibited negligible Joule heating effects with Auger recombination majoring in output power saturation at high pumping currents. Also, a set of 4.2 and 3.4 µm packaged LEDs were fabricated for degradation experiments.

The high refractive index of the InAs-based materials tends cause strong back-reflection from the out-coupling surface, thus reducing efficiency. Therefore, the LED is equipped with a lens attached directly onto the contact-pad-free surface of the chip by the use of high-refractive-index glue. This increases the out-coupling from the chip and thus the efficiency of the LED. In the MINIGAS sensor, the lens is also important in optimising the optical coupling through the sample gas and into the relatively small window of the miniaturised photoacoustic cell. The coupled infrared intensity corresponds directly to the overall sensitivity of the sensor, especially because the output powers of the mid-IR LEDs are relatively low, for instance, in the order of 100 µW for LEDs at around 3.4 µm wavelength.

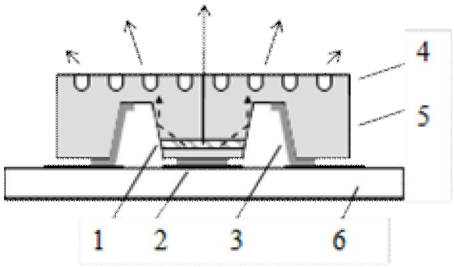

Figure 9. Example of mid-IR-LED architecture. 1 - active area (usually semiconductor double heterostructure); 2 - mirror anode; 3 – cathode; 4 – out-coupling surface with two-dimensional photonic crystal (hexagonal arrangement); 5 - transparent substrate material.

Ioffe developed heterostructure IR-LEDs (emitting at 3.4 µm) both equipped with Si lenses of various sizes and shapes (Figure 10, left) as well as LEDs equipped with micro-immersion lens (Figure 10, right). Si lenses with shape close to hyperhemisphere were attached to the contact-free surface of LEDs by the use of high-refractive-index glue. The LEDs with lenses with diameter of 10 mm with the result that radiation beam narrowed to a minimum at a distance of 25 – 30 mm with the corresponding beam width parameter of 2.5 mm. Moreover, such LEDs were successfully tested by Gasera at a quasi-CW mode (33 Hz) using Gasera's existing commercial gas analyser. Other LEDs were equipped with micro-immersion lenses made from chalcogenide glass (refractive index 2.4). The technology provides high radiation extraction efficiency from the LED together with small source lateral size (<1 mm). This enables achieving reasonable compromise between the extraction efficiency and the coupling efficiency into the miniature PA cells with simple optical schemes. On the other hand, micro-immersion lens LEDs exhibit poor beam quality meaning that high aperture optics is needed for coupling into a sensor, such the PA cell.

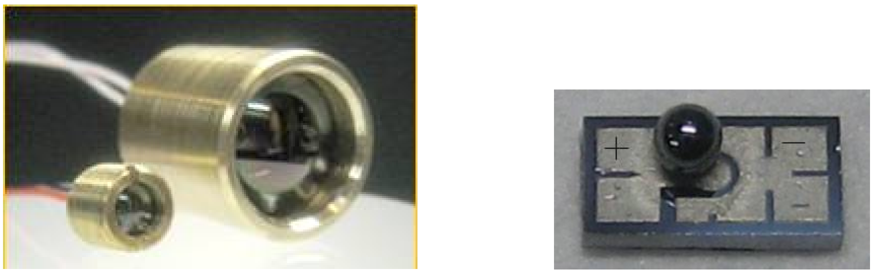

Figure 10. Examples of photos of developed mid-IR-LEDs equipped with lenses: Left) Two LEDs packaged with Si lenses having diameters of 3.5 and 10 mm; Right) LED chip with micro-immersion lens on top and assembled onto silicon submount having dimensions of 2.9 x 1.5 mm.

Packaging improvements, namely several changes in assembling procedures and materials, lead to high fidelity of the mid-IR LEDs together with high performance characteristics. During the MINIGAS project, packages were adapted to the needs of PA measurements. For the gas sensor demonstrator, VTT developed a IR-LED packaging solution in which LED sub-assembly was attached on top of a thermo-electric cooler (TEC) element placed in TO5 can base. The TEC enables temperature stabilization of the LED device required to achieve stabilized optical power from the LED, even with variable ambient temperature. The TO5 cans were hermetically sealed in inert atmosphere.

3.2.5 Initial sensor modelling and experiments

Before implementing the miniaturised sensor dmeontrators, the sensor concept was optimised and its feasibility was evaluated by analytical and experimental modelling.

Analytical modelling of the PA sensor:
The key parameter of the development of the photoacoustic gas detection is the sample cell volume. By decreasing the dimensions of the sample cell the miniaturization of the whole system is possible. A model system of the differential PA cell was simulated in order to find optimal parameters for the cell and cantilever dimensions. First, a physical model was presented including various sources of the noise. The CO_2 gas was used as an example, partly because its characteristics are well known. In order to use very small cell structures, accurate modelling of the basic physical phenomena involved in all photoacoustic mechanisms is needed, including light absorption in the sample gas, thermal properties of the sample cell, cantilever dynamics, and noise characteristics of the system. After all the essential mechanisms are known, realistic simulations can be performed and reasonably accurate predictions can be done. The results suggested that sub-ppm detection limit can be achieved.

Initial evaluation with MIR-LEDs:
Performance of LED-based MINIGAS sensor, i.e. the detection limit of gas concentration, for various gases as predicted by modelling is shown in Table 1. Our initial experiments performed with PA cells equipped with mid-IR LEDs and cantilever microphone showed that the ppm measuring range is achievable for CO_2 and C_nH_m gases with only 1 s integration time. Results are in the same order as the detection limits observed with tunable diode laser sources working in NIR range. The detection limit for methane is 250 times smaller than previously reported, a remarkable improvement.

Table 1. Performance of MIR-LED-based MINIGAS sensor for various gases as predicted by modelling. Detection limits calculated for signal-to-noise ratio of 1, and assuming 1 s measurement time and 500 Hz modulation frequency.

Gas		Peak wavelength of LED [um]	Detection limit [ppm]	Remarks
Methane	CH4	3.4	0.9	
Carbon monoxide	CO	4.7	7.6	
Carbon dioxide	CO2	4.2	0.6	
Hydrogen cyanide	HCN	3.0	11	
Hydrogen fluoride	HF	2.95	18	
Water	H2O	7.0	3.5	
Formaldehyde	H2CO	3.4	0.5	
Ammonium	NH3	5.3	45	
Nitrogen oxide	NO	5.0	27	
Nitrogen dioxide	NO2	5.0	5.0	
Nitrous oxide	N2O	4.7	0.9	
Sulphur hexafluoride	SF6	10	7.6	LED not available
Sulphur dioxide	SO2	7.0	7.0	

Experimental modelling of the spatial interferometer:
Also a large-scale prototype of the spatial interferometer was designed, implemented and tested. The main targets for the interferometer performance were as follows:

- Resolution of the interferometer well below 1 pm with 1 s measurement time

- The maximum allowed relative error in the displacement measurement is 1%, but the performance will be superior if the error is equal or less than 0.1%.

The designed interferometer consists of a focused beam laser diode, an angle mirror which splits laser beam in two and a CMOS linear image sensor.

Experimental modelling of the PA sensor with various sources:
The current status of IR LED technology does not allow for the long wave range of 7 – 10 µm, preventing implementation of sensitive PA sensors for such gases as SF_6 and SO_2 as Air pollutants, and Sarin or simulant gases for Homeland Safety applications, which were among the original applications targets of the MINIGAS project. However, other light sources are available on the range, and their cost/performance trade-offs were surveyed during the project and for some sources were evaluated with the sensor. Therefore, the PA sensor technology developed in the MNIGAS project was made "flexible/modular" meaning that the sensor can work with other light sources already existing at the market or currently developed. For instance, the detection of Sarin or simulant gases would be possible with a Quantum Cascade Laser (QCL) that is tunable with an external cavity at 10 µm. This kind of laser source is still a novel, niche product with small volumes today, from a few suppliers. However, the activies on the development of novel and low cost QCL lasers at IR is very promising and growing in Europe, too.

Experimental verification of the proposed modular setup with LED source for methane (CH_4) and laser source for SF_6 and nerve gas precursor (DMMP) was needed in order to check possible unknown factors on the over-all function of the system. For this purpose, a differential photoacoustic system was built in the laboratory. It should be noted that the PA setup used in these experiments was modified from an earlier large-scale prototype system and therefore it was not very similar, in the terms of essential parameters, as the MINIGAS design.
Signal resolution of laboratory sensor prototype for DMMP (Sarin simulant) was using an external-cavity QCL source, whose output wavelength was widely tunable between 9.1 – 9.9 µm. The experimental detection limit of this not fully optimised sensor setup was determined 0.65 ppb with 0.6 s measurement time.
In the experimental setup for SF_6 measurement, the light source was CO_2 laser (with average output power of 400 – 700 mW and wavelength 10.5 – 10.6 µm) and photoacoustic system had a sample cell with length of 100 mm. The determined detection limit was 0.12 ppm, and tt was estimated that with an optimised system this could be reduced by more than two orders of magnitude (i.e. below 1 ppb).
A broadband black body IR source could be used as an alternative to IR LEDs. The output power of the black body radiator can be high but in practice only a very small fraction of it can be utilized since the emission spectrum is very wide. In addition, the modulation frequency must be low since the thermal capacities limit the maximal pulse frequency. Use of a miniature IR radiator source was evaluated by experiments.

3.2.6 Conceptual design of the miniaturized sensor

The MINIGAS sensor concept developed strongly during the course of the project. The overall 3D integration and miniaturisation of the sensor structure was developing in parallel with the development of the main components and sub-systems, namely the IR light source (LED), the measurement gas cell, the photoacoustic differential cell (with cantilever), the read-out interferometer and the sensor and drive electronics, were developed and their performance modelling and testing was carried out. The most miniaturised concept design developed and based on the sub-systems described in the previous chapters is presented in Figure 11 and Figure 12.

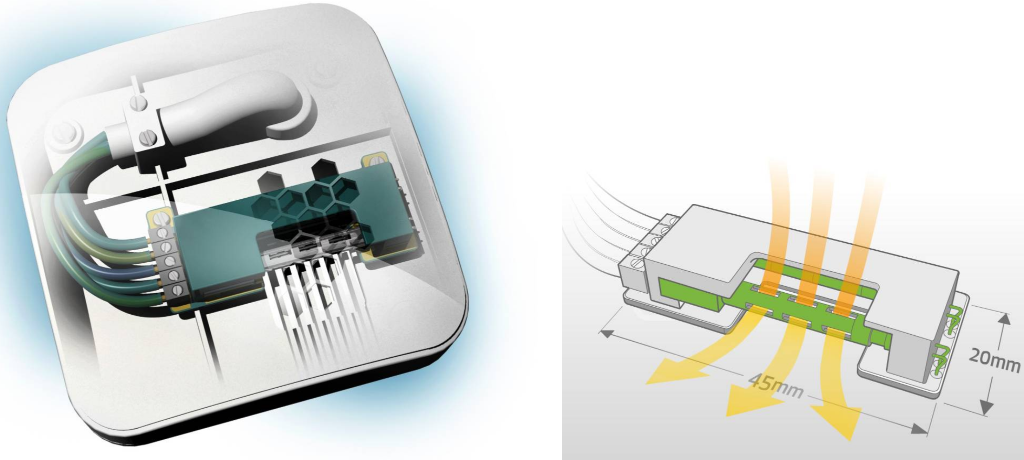

Figure 11. Concept design of miniaturised MINIGAS gas sensor: Left) Complete Gas Analyser in enclosure integrating Photoacoustic Sensor Engine and sensor electronics. Right) Gas flow illustrated through the PA Sensor Engine.

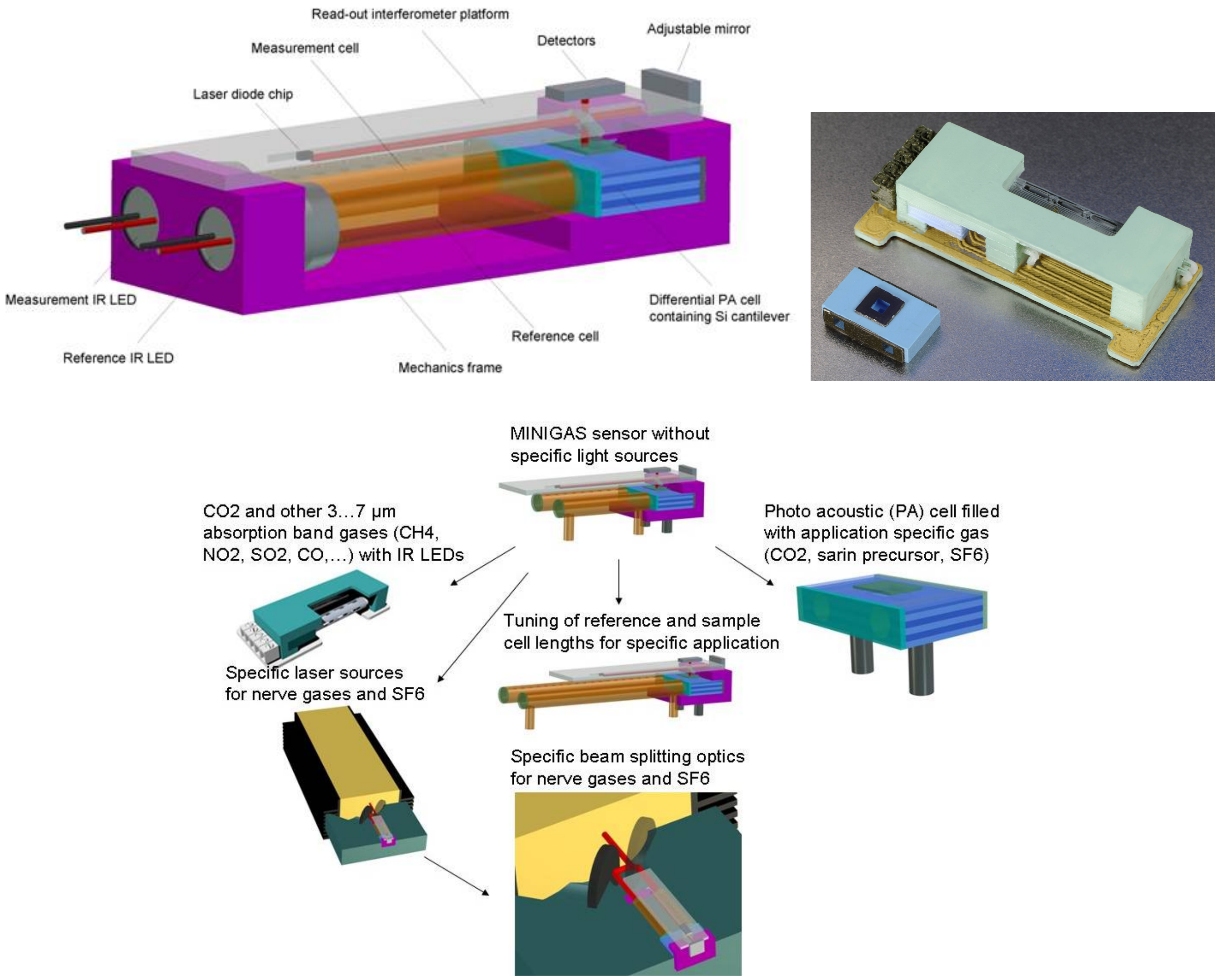

Figure 12. Concept design of miniaturised MINIGAS gas sensor: Top left) Complete Photoacoustic Sensor Engine (volume 5 cm^3) with two IR-LED sources; Top right) Photos of fabricated differential PA cell and mechanical proto (mock-up) of the PA Sensor Engine; Bottom) Modularity of the concept structure (enabling use of various IR sources) illustrated.

3.2.7 LED subassembly and optical coupling

The LED sources are initially well suited to integration within of the MINIGAS sensor. However, the radiation powers of the novel mid-IR LEDs are relatively low still, thus there is no possibility to "waste" a significant amount of power without compromising the sensor performance, i.e. the signal-to-noise ratio. Therefore, high efficiency optical coupling is required from the LED through the sample gas cell (with long enough absorption path) and into the relatively small entrance window of the miniaturized differential PA cell (ca 2.5 mm rectangle). This is a significant challenge for optical design. During MINIGAS project, various optical designs and optomechanical constructions were investigated targeting best compromise between the high coupling efficiency, suitable absorption path length, and miniaturized sensor integration.

Additional design challenge is that the LED subsystem consists of two LEDs, one for the sample channel and the other for the reference gas cell of the differential PA cell. The IR radiation from the sample and reference cell is fed to the chambers of the differential absorption cell. In the case that no absorption occurs in the sample cell, the light intensities in the two chambers of the differential absorption cell should be precisely equal in order that no cantilever displacement occurs. In practice although the LED output intensities can be made equal it is still difficult to guarantee that the light intensities entering each chamber of the differential cell are equal because of: (i) variations in LED beam geometry and orientation, (ii) optical reflectivity of the walls of the sample and reference cells, and, (iii) optical transmission of the windows. Therefore, the output intensities of the LEDs should be adjustable. This can be done by adjusting the current of the LEDs.

Two different kind of potential schemes for optical coupling were identified and studied in detail. In those schemes, as illustrated in Figure 13, the LED for the sample gas channel was equipped with either:

a) Conventional "large" Si immersion lens to enable narrow divergence beam imaged towards the cell window.
b) Small micro-immersion lens (MIL), which should be combined with elliptical/spherical mirror.

LED equipped with Si immersion lens forms magnified image at a distance from LED and thus the LED chip can be imaged to the rectangular window of the differential PA cell through a few cm absorption path. With proper lens diameter it is possible to get an LED image dimensions that match the cell window. The disadvantage of the scheme is that the overall coupling efficiency from the LED chip is only moderate. The overall efficiency may be improved if a cylindrical sample gas cell with high wall reflection efficiency is used (as illustrated in Figure 13 a). Alternative to cylindrical cell, a concave optical concentrator could be used in front of the cell window to enhance coupling in the cell.

On the other hand, micro-immersion lens technology provides high out-coupling efficiency from the LED chip. However, MIL LEDs exhibit poor beam quality meaning that high aperture additional coupling optics is needed for the sample channel. This can be implemented using a spherical or aspherical mirror lens, possibly off-axis mirror. Thanks to the small dimensions of the micro-immersion lens, the LED power can be imaged with high efficiency on the cell window using an optimised mirror lens (as illustrated in Figure 13 b). As a conclusion, the IR-LED equipped with a micro-immersion lens was used as the sample LED in the sensor demonstrator, and a spherical mirror of a short focal length was used to increase the optical coupling efficiency into the PA cell though the sample gas.

Because no gas absorption path was needed in the reference path, the reference LED was selected to be equipped with Si lens, which enables direct coupling into the differential cell with high efficiency with the rather short separation between the LED and cell window (as illustrated in Figure 13).

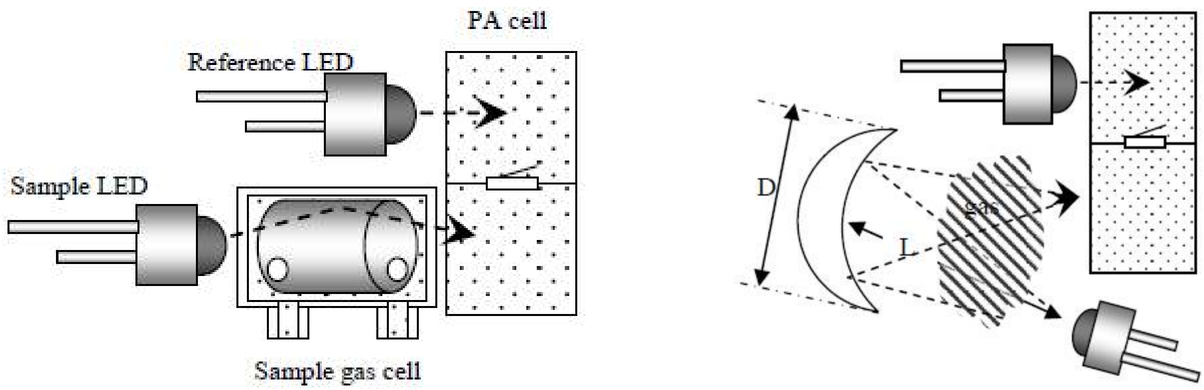

Figure 13. Schematics of the two studied optical coupling schemes: Left) Coupling with LED equipped with large lens (and potentially enhanced with reflective walls of the sample cell); Right) Coupling from LED equipped with microimmersion lens and by the use of spherical mirror lens.

3.3 Handheld methane sensor demonstrator

To demonstrate the concept of the novel miniaturised differential photoacoustic sensor and to evaluate its performance, a handheld prototype of the sensor was implemented and tested. It was built using the MINIGAS components and sub-systems described in the previous chapters, that is, using the mid-infrared LEDs, LTCC-based differential photoacoustic cell, MEMS cantilever and a spatial interferometer. The proof-of-concept demonstrator was integrated into an aluminium optomechanical housing, which produced the verification of the sensor concept essential to continue development towards even a more miniaturised sensor integration platform in the future. In addition, the sensor housing was designed as modular to enable switching the IR LED sources and the measurement cell with a measurement cell optimised for an external laser diode source (with splitted beams), thus allowing also measurements of others gases at longer wavelengths where LEDs are not available yet.

3.3.1 Design and implementation

A functional block diagram of the MINIGAS sensor system is shown in Figure 14. It also depicts the types of the physical interfaces between the parts and sub-systems. (The operation principle of the sensor head was described in Chapter 3.1.1.) The sensor head essentially consists of the IR light sources, the differential cell system, and the readout interferometer. The LTCC-based differential PA cell sub-system contains a reference cell, a sample cell and MEMS cantilever. The cantilever senses the pressure difference between the two parts of the PA cell filled with pure methane, which was the target gas. The cantilever movement is monitored with a spatial readout interferometer. The measurement system also contained interferometer readout electronics, thermoelectric cooler (TEC) controller, and the LED source electronics, which all were controlled with PC software.

The PA gas detection technique was combined with mid-IR LEDs equipped with micro-immersion lenses and operating in the 3.4 µm wavelength range, which covers the absorption bands of many important hydrocarbons (CH_4, C_3H_8…). The selectivity in the demonstrated sensor can be tuned simply by filling the differential cell with specific gas under interest and selecting corresponding MIR LED with proper emission spectrum that cover the selected absorption peak of the monitored gas.

The IR sources were a thermally controlled pair of LEDs. The IR radiation of the other LED is coupled to the PA cell through the open path measurement cell (with free flow of the sample gas), whereas the radiation of the other LED is directly coupled to the reference part of the PA cell. That is, the reference

LED was in close contact to the window of the PA cell. The light from the IR source passing through the measurement cell is attenuated if any of the gas species of interest is present. The calibration of the sensor head, i.e. the cantilever balance in zero concentration of monitored gas, was achieved simply by tuning electrically modulated IR output powers of the LEDs to produce equal pressure increments from the reference and sample cells to the cantilever.

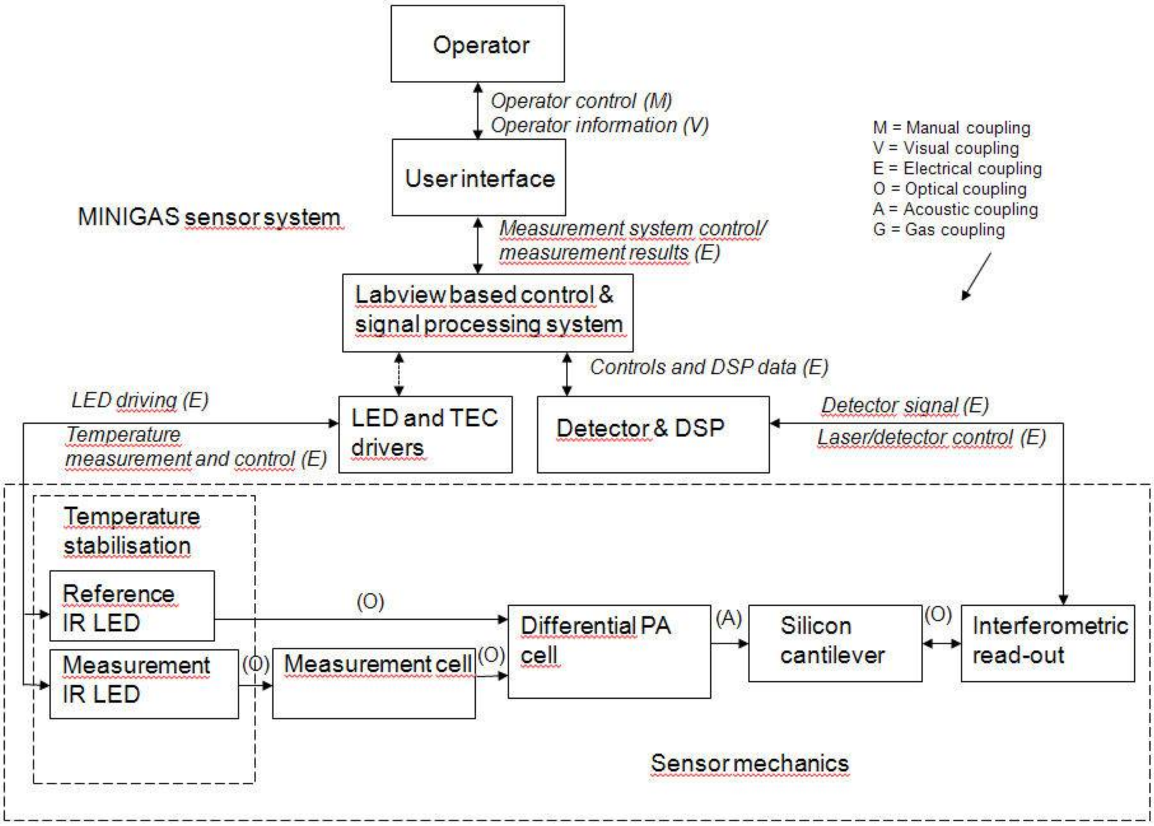

Figure 14. Functional block diagram of the sensor demonstrator. The interface types are indicated with letters.

The integration platform design of the CH_4 sensor prototype was based on optomechanical aluminium housing. Optomechanical housing was tooled using 5-axis CNC machine and it contained support, alignment and attachment features for the individual functional blocks, such as the IR LEDs, the spatial interferometer and its laser diode and CMOS detector on circuit boards. The spatial interferometer was implemented into a small aluminium block using discrete optical and optoelectronic parts. In addition, a spherical mirror for collecting optical power at the open path cell and coupling LED power to the sample cell was aligned and assembled. The heart of the sensing system was the LTCC-based differential PA cell containing monitored gas, which was pure methane in slight over pressure (in between 101–152 kPa).

In the implemented IR-LED based the IR LEDs are temperature stabilised and mounted in TO5 can. The sensor electronics and signal processing, however, is separate from the sensor head and mounted outside the sensor mechanics. The electronics controls current supply to the two IR LEDs and the TEC drivers to temperature stabilise the LEDs and other critical devices. The sensor electronics and signal processing also controls the 650 nm VCSEL-type laser diode in the read-out interferometer and the associated detectors and pre-amplifiers.

The size of the demonstrator is about 40 mm x 40 mm x 35 mm, i.e. much larger than the original project target volume 5 cm^3. The difference is mostly due to the mechanical implementation, and it is assumed that the sensor size could be reduced in a product development project.

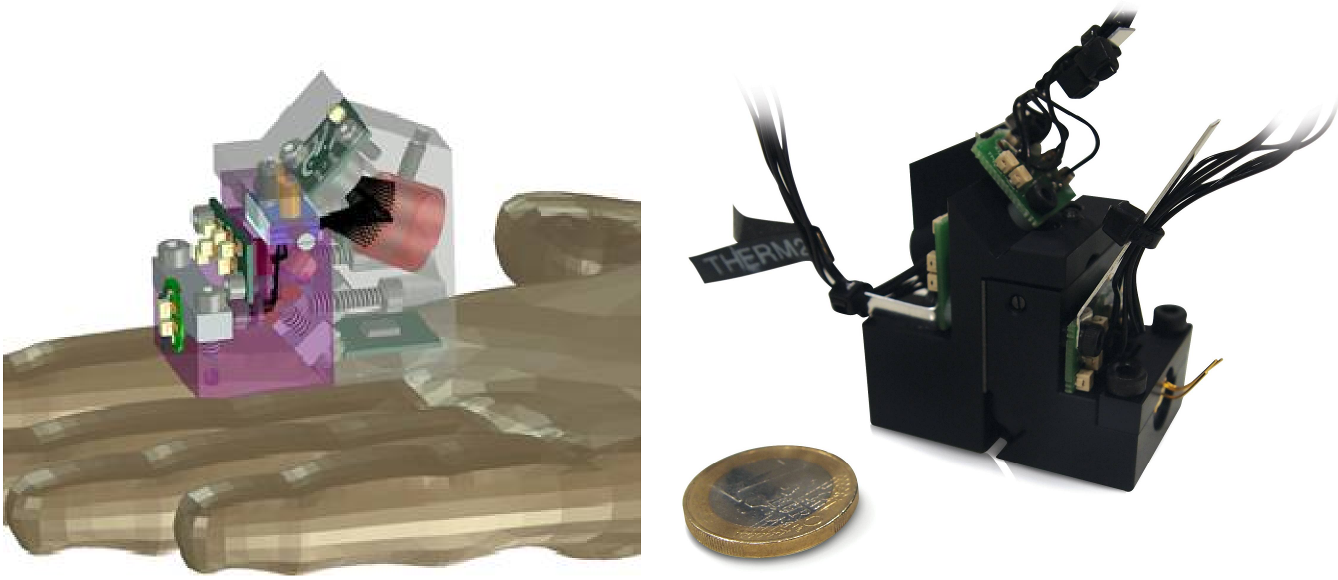

Figure 15. Methane sensor demonstrator; left) Optomechanical design of the sensor head “on palm”; right) Photo of the assembled demonstrator.

3.3.2 Testing and evaluation

The operation and performance of the assembled methane sensor demonstrator was evaluated by placing it in a chamber, in which methane concentration could be adjusted in a large range. The balancing of the optical power in the sample and reference PA cells was achieved in zero methane concentration by adjusting the driving currents of the LEDs. The operation of the spatial interferometer was verified to produce 1 pm resolution, when probing the cantilever tip position. The obtained differential pressure signal was proportional to concentration of (methane) gas flowing in the open measurement path cell. The detection limit of the sensor demonstrator for methane was 110 ppm at 1 x rms (root mean square) noise level with 1 s measurement time, and 330 ppm at 3 x rms noise level. Sensitivity was increased to 30 ppm, when response time of 100 s was used. The signal suffered from a strong drift, which is probably due to different thermal conditions of the LEDs. This indicates that more careful stabilisation of the IR sources is necessary in the future.

Preliminary environmental testing of the handheld CH_4 sensor demonstrator was carried out too. Reference for the test plan was the MIL STD 883 for electronic components, and particularly, due to the civil industrial application target, the EN 600068. However, due to the fact that the demonstrator had not been designed for reliability or for harsh environment only a limited test plan was performed. In particular, the vibration testing was done only to verify the natural resonances of the mechanical assembly of the sensor. Sinusoidal vibration testing (following MIL STD 883H method 2006.1) could be completed without noticeable damage to the sensor. Temperature cycling was performed over the operating temperature range with the device switched on, but only the operation of the interferometer based cantilever readout was verified during the test. In thermal cycling testing the sensor operation at temperature range of -20…+40°C was stable. However, both at low (-30…-20°C) and high (+40…+60°C) temperature ranges component misalignments in the aluminum housing caused signal loss. In conclusion, the environmental test results with the CH_4 demonstrator were encouraging, although some problems arise during testing and especially during temperature cycling in high and very low temperatures. This is not surprising as the demonstrator was a breadboard and its mechanical housing was not specifically designed for wide temperature operation (which however could be done in the future).

The LTCC-based differential PA cell was tested to fulfill MIL STD 883 according to hermeticity. The MIR-LEDs were hermetically sealed, and the window of the reference LED package was engaged directly to the differential cell window in the demonstrator, although this reference path was not hermetically sealed in the demonstrator.
Based upon the implemented sensor system prototype the mass manufacturability of the sensor was evaluated applying cost-of-ownership (COO) modelling. The accuracy of the modelling was hindered by the fact that the demonstrated sensor system is not yet developed for early product level, being rather a laboratory prototype. In addition, there is no test series production experience to give valuable information about accurate yield, utilisation rate and throughput time values.

3.4 Greenhouse gas detection demonstrator

In the other MINIGAS project final demonstrator, the performance of differential photoacoustic setup was demonstrated for greenhouse gas flux monitoring application. The target gases in the application are H_2O (water vapor) and CO_2. The end-users for such an analyser are research groups motivated by the global warming. This is a well-known niche market and end customers have a strong need for improved instrumentation in terms of sensitivity, response time and field portability.

On the component level, the MINIGAS demonstrator for the greenhouse gas flux monitoring application was an ultra-high performance system utilizing tunable IR diode laser sources. In order to demonstrate the technical achievements in the MINIGAS components and subsystems (the miniaturized spatial interferometer, optimized MEMS cantilever, and miniaturized differential PA cell), they were combined with two tuneable IR laser diode sources: one laser diode for CO_2 and the other for H_2O measurement. The idea was to demonstrate similar (or even better) performance compared to industry's most high-end and high-cost technology with the MINIGAS platform, which can be ultimately productized in handheld size and the price would be a fraction from the state of the art systems.

3.4.1 Implementation

For stand-alone operation, the greenhouse gas detection demonstrator was implemented in a rack enclosure used in Gasera's existing analyser instruments. The lasers were coupled to the differential PA cell module and the whole sensor system was mounted in a rack-mount analyser including user interface computer. (Figure 16)

The measurement setup consists of the differential PA cell and compact spatial type readout interferometer. Two commercial diode lasers operating at 1877 nm (for H_2O) and 2702 nm (for CO_2) and packaged in TO5 cans were used. In the measurements, the wavelengths of the lasers were modulated over the absorptions lines of the target molecules. The modulation was possible by adjusting the laser current.
Custom optics and optomechanics were designed and implemented for the laser beam splitting and focusing. The sample and reference cells were in front of the photoacoustic cell having length of 177 mm. The light from the two diode lasers for water and carbon dioxide measurement was focused to the PA cell entrance through the sample and reference cells. The lasers were located on the two arms of the beam splitters that divide the laser beams in to both sides of the cantilever.

In order to realize the laser source based demonstrator some modifications to the MINIGAS components were required:

- The MINIGAS spatial interferometer design was used. However, the interferometer housing had to be redesigned for integration into this demonstrator.
- Cantilever mounting was implemented using O-rings, in order to be more confident about achieving targeted performance for the demonstrator. Consequently, the cantilever frame size was increased, although the dimensions of the cantilever tip itself were kept the same (in the measurements, tip thickness was 4 µm).
- New differential PA cell was needed to enable O-ring mounting of the cantilevers. Because the PA cell size became slightly bigger compared to the original MINIGAS cell design (and due to schedule issues), the cell was manufactured of metal. Metal is applicable choice also since the laser diode -based system is a high performance sensor and the production volumes of such systems are lower compared to low cost sensors; thus LTCC would not provide significant added value in this case.

The photoacoustic cell was filled with 99.9 % nitrogen mixed with water to 500 mbar pressure in the carbon dioxide measurement. In the water measurement, the pressure in the PA cell was 120 mbar and background gas was 50/50 % of Xe/CO_2.

Figure 16. The differential photoacoustic gas analyzer demonstration unit for greenhouse gas detection.

3.4.2 Performance testing

The sensitivity of the MINIGAS differential cell gas sensor demonstrator for greenhouse gas detection was measured. The CO_2 measurements were carried out by varying the sample gas concentration by 20 ppm steps from 360 ppm to 500 ppm. Observation time was 0.1 second for each measurement point. Precision was evaluated as standard deviation of 10 successive points. The achieved precision for 0.1 s observation (i.e. integration) time was 0.51 ppm, for 1 s observation time 0.29 ppm, and for 5 s observation time 0.10 ppm.

Water vapor measurements were made for the four concentrations between 0 ppm and 20 000 ppm. Precision values were measured for each concentration with 0.1 s integration time and for 5000 ppm concentration also with 1 s and 5 s integration times. The measured precisions with 0.1 s integration time in concentrations of 0 ppm, 5000 ppm, 10000 ppm and 20000 ppm were 3.4 ppm, 5.8 ppm, 8.5 ppm and 11.8 ppm, respectively.

Performance measurements with the MINIGAS laser source demonstrator confirm major technological achievements in the spatial interferometer, cantilever sensor and differential PA cell components developed in the MINIGAS project.

The measured performance of the demonstrator (Table 2) was then compared to the datasheet values of a commercially available analyser instrument, which is based on the Cavity Ring Down Spectroscopy (CRDS) principle and seen as the state-of-the-art in terms of sensitivity. The selected reference analyser wass manufactured and sold Picarro Inc. The benefit of differential PA principle compared to CRDS is lower cost, smaller size and faster response time. The results (Table 2) show that already the demonstrator prototype performance was competitive against the best available mature solution in the market place in terms of performance.

Table 2. The measured sensitivity of MINIGAS greenhouse gas detection demonstrator compared to the datasheet specifications of Picarro's commercial Greenhouse gas flux analyzer based on CRDS technology (Picarro G2311-f, data sheet available online).

Gas	Integration time [s]	MINIGAS demonstrator Precision [ppb]	CRDS Precision [ppb]
CO_2	0.1	510	150
CO_2	1	290	150
CO_2	5	100	150
CO_2	180	20	50
H_2O	0.1	3400	6000
H_2O	1	1100	6000
H_2O	5	480	6000
H_2O	180	60	6000

4 Impact, Dissemination and Exploitation

This chapter presents the dissemination activities carried out during the project and discusses the potential impact of the work and the exploitation plans for the results.

4.1 Dissemination activities

Information about the MINIGAS sensor technology, applications and project results was disseminated during the project through various activities. The main dissemination activities are described here. Full list of public presentations about the project results is described in Table 3.

In May 2008, a website with an area for public access was created presenting all the latest advances and publications on MINIGAS. Also a general overview on the project and the technology was given on the public webpage. The website was fully revised in the beginning of 2011 and its address became www.minigas.eu.

Public awareness of the technology was enhanced also through two press releases, one launched in the beginning in the project and the other one at the end of the project. Final press release of the project results, titled “Innovative sensor technology for efficient and portable gas detection devices”, was issued in January 2013.
Moreover, a MINIGAS flyer presenting key facts about the technology and the project was prepared and distributed through several channels, for instance, partners distributed it at their booths at tradeshows and exhibition. The flyer was updated a couple of times during the project.

Technical results were presented in several conferences, for example at Sensor and Test, Mid-infrared Optoelectronics: Materials and Devices (MIOMD-IX, -X), ICAVS, PITTCON, SPIE Photonics Europe and Photonics West and specific end-user tradeshows and exhibitions.
At the exhibition booths, the technology and the project were promoted using posters, component samples, mock-ups and demonstrators of the sensor, and MINIGAS flyers. For instance, the MINIGAS mock-ups and presentations attracted people to visit the exhibition boots of Gasera in various events and provided Gasera the possibility to introduce photoacoustic technology in general as well as to acquire information about potential end-users and customers.

Presentations were also be given at European Commission sponsored events and workshops to ensure awareness of the project's results and cohesion within the European Research Area.

Table 3. List of MINIGAS public dissemination activities.

No	Type of activities	Main leader	Title	Date	Place	Type of audience	Size of audience	Countries addressed
1	*Minigas homepage built*	*VTT*	*http://www.minigas.eu*	*2008, revised 2011*		*Industry, scientific community*		worldwide
2	*Present-ation*	*VTT*	*Concerta-tion meeting FP7 Photonics projects*	*Sep 18-19 2008*	*Barcelona*	*Industry, scientific community, policy makers*	*100*	EU
3	*Press release*	*VTT*	*Miniature gas sensors for detecting greenhouse gases*	*Oct 27 2008*	*Espoo*	*Media*		*America, Europe, Asia*
4	*Exhibition, present-ation*	*VTT*	*Sensor+ Test 2009*	*May 26-28 2009*	*Nürnberg*	*Industry, scientific community*	*7000 (visitors)*	*Europe*
5	*Exhibition, present-ation*	*VTT*	*Photonics West 2010*	*Jan 26-28 2010*	*San Francisco*	*Industry, scientific community*	*30 (audience listening) 18000 (visitors)*	*America, Pacific*
6	*Exhibition, present-ation*	*VTT*	*Photonics Europe 2010*	*April 12-16 2010*	*Brussels*	*Industry, scientific community*	*2150 (visitors)*	*Europe*
7	*Exhibition, presentati on*	*Gasera*	*Pittcon 2010*	*Feb 28 - March 5 2010*	*Orlando*	*Industry, scientific community*	*30 (audience) 16900 (visitors)*	*America, Europe, Asia*
8	*Exhibition, present-ation*	*Gasera*	*Sensor+ Test 2010*	*May 18-20 2010*	*Nürnberg*	*Industry, scientific community*	*7400 (visitors)*	*Europe*
9	*Exhibition, present-ation*	*Gasera*	*Pittcon 2011*	*March 13-18 2011*	*Atlanta*	*Industry, scientific community*	*17100 (visitors)*	*America, Europe, Asia*
10	*Exhibition*	*VTT & Ioffe*	*Photonics Europe 2012*	*April 16-19 2012*	*Brussels*	*Industry, scientific community*	*2030 (visitors)*	*Europe*
11	*Press Release*	*VTT*	*Final project results*	*Jan 22 2013*	*Espoo*	*Media*		*America, Europe, Asia*

Details of the technical/scientific work and results were published in several peer reviewed papers and conference contributions during the project. Total 12 scientific journal papers and 16 scientific conference papers or abstracts were published so far, as listed in Table 4.

Table 4. List of scientific (peer reviewed) publications about MINIGAS results (starting with the most recent ones).

No	Title	Main author	Title of periodical or series	Volume number or Event; and pages	Publisher	Year
1	*Portable methane sensor demonstrator based on LTCC differential photo-acoustic cell and silicon cantilever*	*K. Keränen*	*Europhotonics 2012*	*Book of Abstracts, Europhotonics, PT1-33, 9-12 Sep 2012, Krakow, Poland*	*Epapers. org*	*2012*
2	*Radiation distribution in 3.4 µm immersion lens LEDs in far field*	*N.V. Zotova*	*Journal of Optical Technology*	*Vol. 79(9), p. 571–575*	*OSA*	*2012*
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In the beginning of the project, a MINIGAS User Group was created consisting of a few potential end-user companies of the technology. This was to enhance dissemination of the results to the potential end-user companies and to help identifying the most appropriate application areas. This enabled to get feedback and information about the end-user requirements and plans, thus also influencing the project specifications and preparation of the exploitation plans. Workshops and meeting were organized with the invited end-users.

4.2 Impact

4.2.1 Technological impacts

The value that MINIGAS delivers to the market is the ability to achieve gas detection performance normally associated to expensive high-class instruments in a significantly lower price range but still maintaining the characteristics of small size and ruggedness of the class. The advantages of MINIGAS are optimal in portable and handheld gas detection instruments and miniature fixed detectors for selected chemical warfare agents, carbon dioxide, methane and SF_6 but with the limitation mentioned above.

MINIGAS achievements prove that the photoacoustic gas cell can be miniaturized without compromising the sensor sensitivity. The sensitivity enhancement is achieved with a cantilever microphone system that enables robustness for handheld outdoor use. Moreover, the cantilever-enhanced photoacoustic trace gas detection technique was combined with micro-immersion lens mid-infrared light emitting diodes (MIL mid-IR LEDs) operating in the 3.4 μm wavelength range, which covers the absorption bands of many important hydrocarbons. On the other hand, the evaluation with the MINIGAS laser-source demonstrator confirmed major technological achievements in the spatial interferometer, cantilever sensor and differential PA cell components, showing that already the prototype performance is competitive against the best available mature solution in the market place in terms of performance. Tunable lasers are optimal sources with cantilever-based PA detection. In this combination, the cantilever sensitivity and zero background effect of the PA detection are effectively exploited especially when using wavelength modulation. Moreover, the cantilever technology could be useful for high-end devices with multi-gas measurements.

In MINIGAS project, the proof-of-concept prototype implementation based on aluminium opto-mechanical housing produced verification of the sensor concept. This paves the way towards even smaller sensor by further miniaturisation of the integration platform and the subsystems in the future.

Although the primary target was of MINIGAS project was new sensor technology, the project results also include advances on novel components, especially mid-IR LEDS, and on novel manufacturing technologies for miniaturised devices, having potential impacts on other applications too.

4.2.2 Sensor applicability assessments

In the following, the potential target applications of the MINIGAS sensor identified before and during the project are discussed. Also, the applicability of the MINIGAS technology for those applications is assessed based on the project results and benchmarking them against the existing sensors.

Attractive applications for the MINIGAS sensor using laser source are breath measurements, engine tailpipe measurements in automotive engine development, and greenhouse gas flux measurements in air quality research. Common requirement to these applications is the fast response time (0.1 second or less). Furthermore, instrument size and robustness are critical parameters since for example the need in the engine development is to measure in a moving vehicle. Therefore, clumsy laboratory technology cannot be used. The laser source MINIGAS concept provides solution to these market demands. A special feature in the differential photoacoustic setup is the fast response time.

In health diagnostics, the breath volume of children is very limited and therefore the sample flow rate needs to be small setting limits to the size of the measuring chamber. Also the gas sample has a high moisture content thus preventing the use of several sensing technologies and shortening the lifetime of the ones currently used if the moisture is not properly removed with additional components. It is also recognized that the electrochemical detectors used in breath analysis suffer from problems related to the electromagnetic compatibility of instruments.

Measurement of CH_4 with handheld devices (“sniffers”) for natural gas leakage detection was an original application target proposed by the partner Dräger. A new “sniffer” based on MINIGAS sensor technology using LED sources promises significant improvements from the current state-of-the-art devices, including ruggedness, power consumption, response time, discrimination (between natural and marsh gases), lifetime, and calibration intervals. Nevertheless, it appeared the achievable detection limit and stability of the CH_4 sensor demonstrator using MIR LEDs as light sources does not meet the requirements for a natural gas leakage sniffer, and it is unlikely that technology would allow meeting the requirements in the near future. However, if a laser diode was used as excitation source, it is very probable that the MINIGAS sensor could reach ppm-level sensitivity for CH_4. Unfortunately a laser with some mW emission power requires temperature stabilization and special measures for protection in explosion hazardous areas. This would add additional manufacturing costs to the system. Existing commercial sniffers reach 1 ppm sensitivity. As a disadvantage, they can be poisoned and need calibration with a test gas every few hours. However, they can be manufactured at much lower costs than a MINIGAS sensor equipped with an expensive laser source could be, thus it seems not probable that the MINIGAS sensor could become competitive as a sniffer in the next few years.

Greenhouse gases are gases in an atmosphere that absorb and emit radiation within the infrared spectrum. This process is the fundamental cause of the greenhouse effect. The most abundant greenhouse gases are: water vapour, carbon dioxide CO_2, methane CH_4, ammonia NH_3, nitrous oxide N_2O, ozone O_3 and the halogenoalkanes CFCs. Legislation is one of the main driver enforcing new emission gas sensors for the markets. The application aimed with the MINIGAS laser sensor was high-end analyzer in the field of greenhouse gas flux measurements. In the demonstrator, the MINIGAS differential PA cell was coupled with near infrared tuneable diode laser sources for the simultaneous measurement of atmospheric carbon dioxide and water vapour concentrations.

Sulphur Hexafluoride (SF_6) is used in the power utility industry as an insulator mainly for electrical switch- and control-gear. Unfortunately, SF_6 is one of the most potent greenhouse gases with a Global Warming Potential 25000 times greater than that of CO_2. There are no indications that SF_6 can be replaced with another material in the foreseeable future, rather, the use of SF_6 is increasing. Therefore, continuous monitoring of SF_6 is favourable and may even become required by law in the future. Due to the lack of feasible LED at >7 µm wavelengths, the PA sensor is applicable only if a laser is used as an excitation source which unfortunately increases the size, power dissipation and costs of the sensor.

The increasing threat of international terrorist attack is driving a large demand for devices with high sensitivity and enhanced selectivity in sensing of a large class of materials, such as, gases used as biological weapons and representing a threat in case of vapours from explosive compounds. The original MINIGAS application scenario proposed by the partner Selex aimed to a high sensitive device able to detect some selected toxic chemical weapons. The new photoacoustic sensing technique should be characterised by low size and weight detectors for security and terrorist attack countermeasures applications. In homeland security, a small number of chemical sensor or bench-sized spectroscopy systems are well established in the field, all of which suffer from significant problems in throughput, specificity and detection level (chemical sensors) and, generally, environmental ruggedness. Semiconductors are lacking selectivity, especially when monitoring nitrogen oxides, and electrochemical sensors need improvement in sensitivity. The miniaturized PA sensor is an attractive solution if combined with the emerging tunable quantum cascade laser diode source at ca 10 µm wavelength range.

4.2.3 Exploitation plans

The gas sensor market analyses indicate strong demand for handheld and portable gas sensing devices with high sensitivity, as was presented in Chapter 2.1.1. MINIGAS project results are paving the wave towards an ultra-sensitive miniaturized gas sensors and analysers. A long-term vision of such a handheld mobile analyser by Gasera is shown in Figure 17. Features of the product vision by Gasera include:

- Handheld multi-gas analyser
- Based on infrared photoacoustic spectroscopy
- Simultaneous analysis of hundreds of toxic gaseous compounds
- Sub-ppm detection limits in one minute

- Mobile phone size
- Utilizes the ultra-sensitive cantilever sensor

Figure 17. An artistic vision of a handheld mobile gas analyzer based on photoacoustics (by Gasera).

Performance measurements with the MINIGAS demonstrators confirm major technological achievements in the developed spatial interferometer, cantilever sensor, differential PA cell and mid-IR LED components. On the other hand, the applied manufacturing technologies enable significantly reduced volume production costs compared to the competitive sensors on the market. Moreover, the results with the laser source based demonstrator already showed competitive performance against the best solutions available in the market.

The aims of the MINIGAS project included the preparation of an exploitation plan that will formulate the strategy for transitioning the gas sensor into the market place within a following few years. The consortium partners targeted commercialization of the developed sensors or licensing the IPR generated throughout the MINIGAS programme. To guide the consortium towards this goal, an Exploitation Committee (EC) was formed during the early stages of the project, with membership comprising of at least one representative from each of the consortium partners. The MINIGAS competitive positioning was evaluated by the partners. Among others, the previous data of the market profile (size, competitors, stage of growth), customer segments (groups of prospects with similar needs) and other exploitation potential analyses were utilized.

The industrial partners are considering products based on the new technology. Furthest advanced is Gasera Oy, which already uses the parts developed in the project in its current gas sensors. The company hopes to be selling new products based on the technology within three years. Measuring instrument companies Selex and Dräger, as well as LED component developer and manufacturer IoffeLED, plan to use or further investigate parts of the developed technologies for future products.

Gasera’s business model is to launch new products in cooperation with large international companies in most of the market segments, such as the end-users in this project. Gasera has already exploiting the results of the MINIGAS project in the component level. MINIGAS also provided a platform to further develop the novel spatial interferometer by Gasera. Thanks to these achievements the spatial interferometer component has been implemented in all current Gasera products and project prototypes. Furthermore, for Gasera, an important achievement of MINIGAS is the networking, which is vital for the future development and manufacturing and has already led to several new projects where the core components of the photoacoustic system are utilised or further developed.

Mid-IR LED and photodiode manufacturer Ioffe will continue collaboration with the instrument companies that produce IR gas analysers in Russia and abroad. Currently, the applications include breath analysis and moisture measurements in air. New areas of applications are expected to arise with the micro-immersion lens technology that will be introduced into production soon. IR LEDs could substitute

some of the conventional thermal light sources (filaments, thin film emitters) in optical gas sensors due to the mechanical robustness and lower power consumption of the LEDs. Potential applications are seen among others in the medical, safety and process engineering markets. New customers are expected via participation at the international conferences and exhibitions and through collaboration with the device distributors in Europe. Performance and reliability improvement as well as manufacturing cost reduction can stimulate new instrument designs with subsequent increase of the production volumes. Overall high brightness LEDs with low temperature drift are the preferred for sensors.
Moreover, the LED sources may also find applications in other than the gas analysis areas, for example, in thermal imaging. The development of such thermal imaging systems call for the necessity to create devices that can be used for system calibration and continuous monitoring of detector elements. Linear LED arrays are an example of such devices.

Along with the development and marketing of the LED-based systems, Ioffe LED and Ioffe Institute will continue developing of photodiode based analyzers that utilize photodiodes with layer arrangement (heterostructure type) similar to LEDs. Simultaneous research work on both LED and photodiodes will decrease the development period and costs since many features and technological processes are similar for both LEDs and PDs.

Some MINIGAS results are exploitable also in much wider area than just the IR sensors. For instance, MINIGAS has enhanced VTT's technology portfolio and competences related to:

- multi-disciplinary 3D integration and packaging using LTCC technology,
- implementation of optical sensors, in general and especially photoacoustic gas sensors,
- fabrication of MEMS cantilevers,
- implementation of miniaturised interferometers,
- use of mid IR LED sources.

VTT will continue to promote turning also these research results into new industrial production in the European Union.

Laboratory of Optics and Spectroscopy at the University of Turku (UTU) has gained the scientific knowledge on cantilever-based photoacoustic gas detection methods during the course of MINIGAS project. UTU is the only research laboratory, which has developed interferometric cantilever microphones. The physical model of the system has been improved and especially the part of Brownian noise has been remodelled. The noise physics is a key feature of the model when estimations on the detection limit are done. Also a new interferometric readout system for the cantilever was developed. All this knowledge might be used in other systems where robust miniaturized interferometer is applicable, such as, audio systems, pressure sensor and vibration analysis devices.

4.2.4 Potential socio-economic impact and wider societal implications

Obviously, the applications of the novel sensor technology on air pollution monitoring, industrial safety, homeland security, and healthcare instruments promises to enable significant societal impacts on the development of well-being and on the protection of the environment.

In comparison with the situation before the project, MINIGAS contributed to the increase in employment in small and medium-sized enterprises, especially at Gasera on the PA sensor technologies and at IoffeLED on MIR-LED devices.

Public awareness of the PA sensors and more widely the gas sensor technology and its applications were enhanced through the website and two press releases.

5 Contact details

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