Contenido archivado el 2024-06-18

Nanotechnology based intelligent multi-SENsor System with selective pre-concentration for Indoor air quality control

Informe

Información del proyecto

SENSINDOOR

Identificador del acuerdo de subvención: 604311

Sitio web del proyecto

Proyecto cerrado

Fecha de inicio
1 Enero 2014

Fecha de finalización
31 Diciembre 2016

Financiado con arreglo a
Specific Programme "Cooperation": Nanosciences, Nanotechnologies, Materials and new Production Technologies

Coste total
€ 4 619 315,60

Aportación de la UE
€ 3 399 995,00

Coordinado por
UNIVERSITAT DES SAARLANDES
Germany

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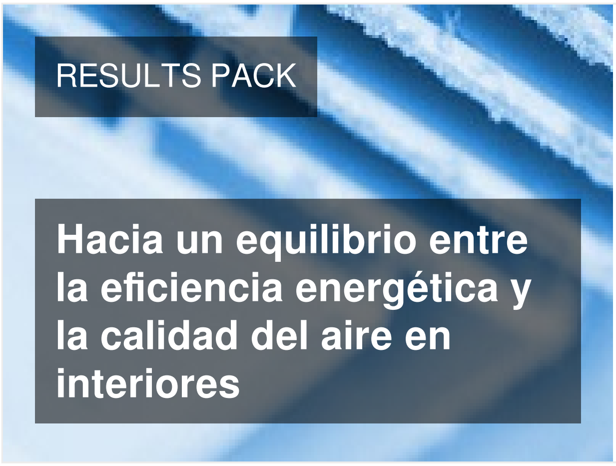

RESULTS PACK

Hacia un equilibrio entre la eficiencia energética y la calidad del aire en interiores

4 Diciembre 2015

Final Report Summary - SENSINDOOR (Nanotechnology based intelligent multi-SENsor System with selective pre-concentration for Indoor air quality control)

Executive Summary:
Closed windows can reduce a building's energy consumption, but this may result in adverse health effects for those who live and work inside them. This is due to the build-up of chemicals from furniture, carpets, paints and cleaning agents. The solution lies in a cost-effective, intelligent ventilation system that automatically supplies fresh air to individual rooms as and when needed, which can be adapted to specific locations such as offices, schools, hospitals or private homes and even specific rooms.
This challenge was taken up by the project SENSIndoor (Nanotechnology based intelligent multi-sensor system with selective pre-concentration for indoor air quality control). Project partners have successfully developed a nanotechnology-based microsystem for selective monitoring of hazardous VOCs to allow demand-controlled ventilation in indoor environments.
The highly sensitive system can detect hazardous VOCs, primarily benzene, formaldehyde and naphthalene at parts per billion (ppb) concentration levels in indoor air selectively against a complex background of other organic and inorganic gases. This was achieved by novel technologies for gas-sensitive layers deposited by pulsed laser deposition (PLD) on two microsensor platforms: metal oxide semiconductor and silicon carbide based gas-sensitive field effect transistors.
These sensors were combined with selective pre-concentrators based on metal-organic framework (MOF) materials deposited on micro hotplates, which absorb the target gases. The integration of novel gas

sensors and pre-concentrator in one microsystem achieves unprecedented levels of sensitivity and selectivity. Novel models for dynamic sensor operation and strategies for application-specific optimization of temperature cycled operation (TCO) were developed to optimize the sensitivity and selectivity towards the target VOCs. Extensive simulations helped to optimize the package layout and the operation for the novel pre-concentrator concept to boost sensitivity and selectivity further.
The technologies were tested in extensive lab tests based of defined target gas conditions and complex variations of interfering gases. In parallel, calibration strategies and methods were developed for factory calibration allowing cost efficient calibration of thousands of sensor systems in parallel with complex test profiles. This is extended by a novel approach for field calibration of the sensor systems based on a two-phase equilibrium of target VOC in a low volatile solvent.
Finally, the sensor systems were tested in realistic field test scenarios both in schools and in private homes. Here, we could demonstrate that the systems can detect if the concentration of one or more hazardous VOC is above a specified limit. In future applications, this would automatically activate the ventilation system to introduce fresh air and to reduce the exposure, thereby ensuring good air quality.
Scaling-up all the technologies involved, especially deposition of the gas-sensitive PLD layers and the calibration of the sensors, has demonstrated the potential low-cost of the developed systems and thus their commercial viability allowing air quality sensors to be installed in each room for comprehensive control of ventilation processes.
Beyond the original scope of SENSIndoor, the project partners have also worked together with other projects, e.g. in the field of metrology. The purpose was to address the need for standardisation in VOC measurements and to establish reliable standards and benchmarks for comparison of different sensor solutions. These standards are essential for users who cannot check the claims made by manufacturers. They are also an improvement on other sensors that are on the market that monitor indoor air quality with a sum parameter for VOCs only. The SENSINDOOR technology can distinguish between hazardous and benign VOCs.
The technologies developed within SENSIndoor for selective measurement of gases in the ppb range will also have an impact in the fields of food safety and health, such as for the diagnosis of acute diseases and for cancer screening. They will also improve industrial safety as benzene is a major concern and its threshold limit value is currently being drastically reduced. In addition, these technologies have security applications for detecting explosives in public spaces such as train stations, airports and marketplaces. The SENSIndoor results will therefore contribute to both the health and security of EU citizens as well as ensuring greater comfort in the home and workplace.

Project Context and Objectives:
Air pollution is still one of the biggest health concerns in Europe and worldwide. Recent studies estimate that more than half a Million premature deaths in Europe are caused by air pollution (Air Quality in Europe — 2015 report, European Environment Agency). The European Environmental Agency (EEA) states in its recent report “The European environment – state and outlook 2015 (SOER 2015)” that while the concentration of most health relevant pollutants is decreasing across Europe, this does not mean that everything is fine: the critical pollutants carbon monoxide (CO), nitrous oxides (NOx), ozone (O3), sulphur dioxide (SO2) and volatile organic compounds (VOCs), especially benzene, formaldehyde and benzo(a)pyrene as well as lead and fine particles still cause serious health defects and far too many premature deaths. This concerns outdoor air, but also indoors as we spend up to 90% of our time inside (SOER 2015), i.e. at home, in the workplace, in kinder¬garten, school or university, in public buildings and

in transport (cars, trains, trams, busses, airplanes). The EU project ENVIE already stated in 2009 “2 Mio healthy life years are lost every year in the EU due to indoor exposure”. Air pollu¬tants lead to “asthma and allergic symp¬toms, lung cancer, and other respiratory and cardio¬vas¬cular diseases”; as well as other types of cancer and even diabetes (Hansen, A.B. et al. (2016), Environment International, 91, 243-250). Air pollutants not only lead to serious health effects, they also impact on our quality of life and affect our cognitive functions resulting in enormous social costs due to reduced productivity in companies (Allen, J.G. et al. (2016), Environ Health Perspect. 124:805–812) and impairs the ability of pupils and students to learn effectively. Young people are especially vulnerable to air pollution. Further aspects of air pollution are the impact on fauna and flora as well as on our infrastructure, i.e. buildings, bridges etc.
One recognized way to address this problem is demand-controlled ventilation (DCV) based on ubiquitous sensor systems that detect and quantify harmful pollutants to reduce their concentration to acceptable levels. To achieve this, low-cost sensor systems are required capable of detecting the primary target VOCs formaldehyde, benzene and naphthalene, which are not only hazardous, i.e. proven or suspected to be carcinogenic, but which also occur in numerous indoor environments thus given cause for health concerns.

Within the SENSIndoor project novel sensor systems for extremely sensitive, highly selective and long-term stable operation were studied and developed for advanced control of Indoor Air Quality. The project made use of both physical and chemical nanotechnologies for sensor components, MEMS technology for component realization and system integration as well as advanced signal processing and networking to integrate sensors into building control systems.

SENSIndoor has achieved this overall aim by realizing the following five specific objectives:
1. Identification of priority application scenarios for DCV based on comprehensive IAQ assessment.
2. Nanotechnology based Metal Oxide Semiconductor (MOS) and Gas-sensitive Field Effect Transistor (GasFET) sensors as complementary sensor technologies with unrivalled sensitivity for hazardous indoor air pollutants, especially VOCs.
3. Nanotechnology based selective pre-concentrators for boosting sensitivity and selectivity by at least two orders of magnitude.
4. Optimized dynamic operation of gas sensors and pre-concentrators combined with advanced data evaluation to further boost selectivity and improve long-term stability.
5. Integration of sensors and pre-concentrators in complex multi-sensor systems and demonstration of their performance in lab and field tests.

SENSIndoor was unique with its strong focus on selectivity. The key to successful development of sensor systems for control of IAQ lies in achieving supreme selectivity to distinguish target VOCs at very low concentrations against a large and varying background of non-toxic VOCs. This selectivity was previously only possible with laboratory equipment such as GC-MS, which is not suitable for control purposes due to high cost and long measurement intervals. While other projects have focussed on sensitivity alone, we have demonstrated ppb-level sensitivity combined with unprecedented selectivity by combining pre-concentration of target gases and temperature cycled operation of the gas sensors. The project has achieved a novel integrated multi-sensor system with unrivalled performance tested in field evaluations for IAQ control.

The SENSIndoor architecture

The project has first identified typical relevant scenarios for IAQ sensor systems, e.g. offices, schools, hospitals and private homes, and defined the requirements in terms of target gases and their concentrations as well as ambient conditions and interfering gases. Due to their health impact and frequent occurrence, the partners have decided to focus on formaldehyde, benzene and naphthalene as primary target gases within the project. Based on this first step, the project addressed the development of sensor systems adapted to specific application scenarios. This was necessary from a scientific point of view to be able to focus on a limited number of scenario specific target molecules with corresponding concentration limits in order to manage the huge spectrum of relevant VOCs, both as target gases and as interferents. In addition, identification of relevant scenarios also allowed us to focus on those scenarios that offer the highest potential from an economic point of view. The most relevant scenarios and the corresponding relevant VOCs including the target concentrations and interfering gases were identified together with the advisory board comprising health standard representatives and major stakeholders from the HVAC (heating, ventilation, air conditioning) industry. This allowed the project to take into account the latest know-how in relevance and impact of various VOCs as well as industrial developments in the HVAC industry in general.

Pulsed Laser Deposition (PLD) was chosen for deposition of novel nanocrystalline materials to achieve highly porous gas sensitive layers. PLD was chosen for its flexibility for deposition of a wide range of different materials, especially metal oxides (SnO2, WO3, VxOy) and noble metal catalysts (Pt, Ir, Pd). Good adhesion of the deposited layers, especially on smooth silicon-based surfaces, and excellent control of parameters such as grain size, layer thickness etc. are further advantages of PLD. Finally, PLD fits well to the technology spectrum for microsensors allowing cost-efficient and reliable mass production of sensor elements. With this approach, gas sensitive layers offering unrivalled sensitivity were developed by optimizing the materials, material combinations and layer deposition parameters. The deposition process was optimized in terms of repro¬ducibility and long-term stability as well as versatile integration of gas sensitive layers on microstructured sensor platforms. Up-scaling of the PLD process to wafer level deposition was demonstrated proving the suitability for low-cost mass production of the novel sensor elements. Efficient testing and calibration of the sensor elements for low-cost mass production was also addressed, both at the sensor element and at the system level.

Preliminary tests in the frame of the proposal preparation with WO3 layers deposited by PLD at the University of Oulu and tested at Saarland University resulted in a relative signal change of 65% for 25 ppb benzene and 900% for 1 ppm ethanol, i.e. the relative sensitivity to benzene is approx. 3 times higher than for ethanol and even in these first tests a detection limit of approx. 2 ppb can be expected (with a signal change of 5%). This compares favourably to the benchmarked commercial VOC sensors that showed a signal response of 20% for 25 ppb benzene and a similar cross-sensitivity vs. ethanol. These first test results provided the basis for further development of PLD deposited layers within the project with the goal of increasing the sensitivity by at least one order of magnitude and also improving selectivity to at least 10:1 by making use of the potential for layer optimization with state-of-the-art ns and ps PLD processes. We also demonstrated a detection limit of 1 ppb for benzene on SiC GasFETs with porous Pt gate layer, indicating that both sensor principles achieve the targeted detection limits under well-controlled lab conditions.

Two sensor technologies, i.e. MOS resistive and GasFET capacitive sensors, were addressed within SENSIndoor because of their complementary detection principles. While the interaction between gas and sensor surface on MOS sensors is mainly due to chemisorption and reaction leading to a charge transfer into the sensor layers, GasFETs can also detect physisorption of polar molecules leading to a surface charge. Thus, both sensor principles together offered a wider detection spectrum plus potential for application specific optimization. Manufacturing for both types of sensors is based on proven semiconductor processes allowing for cost-efficient mass production and allows to make use of the newly developed PLD deposition for both sensor principles. Finally, both sensor principles have shown the potential for improving selectivity by dynamic operation and are also proven to be long term stable, especially in indoor applications.

MOFs (metal-organic frameworks) and, to a lesser degree, MIPs (molecular imprinted polymers) were tested as nanotechnological layers for the realization of novel low-cost pre-concentrators to further increase the selectivity and the sensitivity of the sensor. Micro-fluidic pre-concentrators requiring pumps and valves lead to complex, unreliable and costly systems, while MOF/MIP layers can be integrated on microheaters to achieve very simple, robust and elegant system solutions for at least partially selective adsorption of target VOCs. MIPs are highly cross-linked polymers synthesized in the presence of template (target) molecules. Subsequent removal of the template reveals binding sites retaining affinity and selectivity for the template. MIPs can therefore provide high sensitivity and selectivity similar to antibodies but, unlike their biological counterparts, maintain excellent thermal and mechanical stability. Coating techniques were previously established at FhG-ICT to produce thin MIP layers, e.g. on gold electrodes of quartz crystal microbalance sensors. MOFs have been previously investigated as adsorption materials for small molecules (hydrogen, methane). MOFs are extended crystalline structures composed of metal- or metal cluster-cations ("nodes") and multidentate ionic or neutral organic molecules ("linkers"). MOFs can be tailor-made for a vast diversity of problems ranging from gas separation and storage to the controlled release of guest molecules and their selective inclusion and were demonstrated as suitable material for VOC pre-concentration for low-cost sensors. In contrast to existing techniques, MOFs/MIPs were not used as gas sensitive layer on mass-sensitive devices due to the limited sensitivity of this approach. Instead, target gas molecules are collected by the layer over a longer period and thermally released with a short temperature pulse for the detection by nearby nanosensors. This novel pre-concentrator concept thus improves both selectivity, because of custom-designed molecular adsorption sites, and sensitivity, because of increased concentration of target gases after pre-concentration.

Microcomponents were optimized for the integration of nanotechnological layers for integrated sensor systems, e.g. nanosensors and pre-concentrator platforms placed in close vicinity, placed side-by-side in a common package at a distance of only a few mm. A suitable integration and packaging approach was developed for combining the novel pre-concentrator technologies with gas sensors in low-cost packages suitable for mass production. In these systems, temperature modulation, e.g. of micro-machined membranes with integrated heaters, are used for effective pre-concentration of target molecules (adsorption at RT, desorption at elevated temperatures). In addition, sensor temperature modulation can be employed to increase the selectivity of gas sensor elements by recording the sensor response pattern during temperature cycling and of the pre-concentrator by observing the required desorption temperature for maximum sensor signal of specific molecules. In the SENSIndoor project, SiC GasFETs with nanostructured gate layers were used for the first time specifically for sensitive and selective VOC detection. SiC-GasFETs allow temperature cycling and long-term stable operation by periodic out-gassing at high temperature. MEMS technology reduces the required heater power for sensor and pre-concentrator elements typically down to only a few tens of mW to reach the required maximum operating temperatures allowing low power operation especially in pulsed mode operation. In addition, MEMS components also allow fast temperature changes (typical thermal time constants of micro-hotplates are on the order of a few tens of ms), e.g. for fast temperature cycling of the gas sensors as well as for fast desorption of the collected gas molecules from the pre-concentrator.

Advanced strategies for sensor operation and data evaluation were developed in order to fully use the potential offered by the nanosensors to achieve superb sensitivity, selectivity and long-term stability. Dynamic operation of gas sensors is a powerful tool for increased selectivity by detecting gas specific patterns and evaluating these with pattern recognition tools. The project has concentrated on temperature cycled operation (TCO) a proven approach both for metal oxide sensors and GasFETs in complex environments. As sensor signals are highly temperature dependent, transient temperature changes provide information to discriminate VOCs and measure their respective concentrations. The TCO method was optimized for recognition and quantification of VOCs against a complex background of organic and in-organic interfering gases. The combination of pre-concentration, i.e. variable release of adsorbed gases, and TCO was addressed within SENSIndoor for the first time. The complementarity of the two sensor technologies, MOS and GasFETs, together with the additional information achieved by the use of the pre-concentrator device has provided a unique parameter space. By using advanced evaluation algorithms for the data analysis, the information obtained reliably with this complex sensor system is expanded tremendously compared to existing solutions.

Complete sensor systems were realized, combining nanosensors, pre-concentrator and electronics for dynamic operation and advanced signal evaluation. Sensor systems were evaluated in lab tests with specialized test set-ups to reliably achieve very low VOC concentrations together with background gas compositions under varying ambient conditions, i.e. temperature and humidity. Finally, specific tests in controlled environments reflecting application scenarios were performed to evaluate the performance with controlled release of target gases as well as interferents.

Project Results:
The SENSIndoor project adopted a systematic approach, supported by a well-defined work plan. The work plan comprised ten carefully defined and strongly interlinked work packages. The results presented here are based on these work packages addressing the complete technology chain from definition of application requirements up to final field testing, dissemination and project management.

WP1: Definition of project priorities and requirements

Objectives of this WP
• To identify and rank relevant application scenarios for VOC indoor air quality monitoring in terms of health, comfort, energy saving and economic impact
• To define target VOCs and typical interfering gases, ambient conditions as well as relevant concentration ranges for priority scenarios
• To compile existing national and European regulations on indoor VOC
• To define test schemes, sensor systems components and system architecture for application specific VOC gas sensor systems

Summary of significant results
Priority application scenarios and scenario specific VOC and interferents were successfully identified. There were many interferents identified: Various fat acids from perspiration, NOX and ozone from outdoors, perfumes, chlorine and other cleaning agents. It proved to be impossible to test all arrangements so simplification had to be done according to the most relevant and realistic scenarios.
From these scenarios USAAR-LMT planned the realization of test gases and determined the feasibility of the test. The testing scheme is threefold. The component manufacturers select the most promising samples through a screening test with low complexity. The sensors are then tested with a comprehensive test with application realistic concentration range of 1st priority target VOCs and interferents, which were defined in D1.1. The sensor systems are tested with 1st and 2nd order target and interferent gases.
USAAR-LMT identified methods for generation for all target and interferent gases. The feasibility of generation of some of the target gases (benzene and formaldehyde) has been proven by feasibility tests. For other target gases and interferents the gas mixing system at USAAR-LMT was adapted and upgraded significantly. Tests of feasibility were performed until end of M11.
For the tests performed at USAAR-LMT, NanoSense, with 3S and USAAR-LMT inputs, specified a test bench board able to control various sensor heaters (MOX and SiC) and read out the sensitive layers. This test bench board should also be able to drive a pre concentrator with synchronisation with a sensor. The test bench board should also be able to control the heater in power (considering the ambient temperature and air flow as reproducible and controlled). The PCO (Power Cycled Operation) and TCO (Temperature Cycled Operation) should be easily and graphically settable thanks to a java based software running on a PC. This software should also allow data logging and time stamping of measured heater temperatures and raw read out data.
For the final product the choice of the MCU with co-processor able to handle fast control loop of the heater and read out has been recommended. This is a major input for WP6.

WP2: PLD deposition process for nanostructured gas-sensitive layers

Objectives of this WP
To develop and optimize the Pulsed Laser Deposition (PLD) process onto Si-based micro-hotplates as well as onto GasFET-type substrates in order to get highly sensitive and selective sensing layers via a cost effective and mass-production-compatible fabrication route. PLD method with UV-Excimer nanosecond laser will be utilized to manufacture nanostructured entities of materials, for example ZnO, SnO_2, WO_3, V_xO_y, as well as catalysts, e.g. Pt or Pd, in the forms of nanoparticles, nanowires, and thin films utilizing novel properties of complex structures including phase transitions and heterojunction of mixed phases.

Summary of significant results
Metal oxide sensing layers, with and without metal catalyst, have been successfully deposited on micro-hotplates and SiC GasFET platforms provided as planned by SGX and SenSiC, respectively. Both the nanosecond laser from OU-FETF and picosecond laser of Picodeon have been used in the depositions. Also, tests of PLD depositions with lithography masking have been performed on the MEMS micro-hotplates and the SiC GasFET platforms. Both the use of photolithographic patterning with photo-resist and the subsequent lift-off procedure for restricting the deposition area has been shown possible for low temperature deposition of WO_3. Dicing process of the MOS platform sensor chips with the gas sensitive

PLD layer already deposited has been shown to have no effect to the performance of the sensor. Studies of the scale-up work for wafer level PLD depositions have been performed.

PLD deposited layers

MOS sensors: The porous nanoparticle layers have been successfully deposited on existing SGX platforms by both OU-FETF and Picodeon. A clear dependence of the particle/agglomerate size and also in the level of porosity to the deposition parameters has been recognized. Also, it has been shown that the deposited layer will have a very unique morphology depending on the material which was used. The samples prepared to SGX platforms have been shown to be viable and testable sensors with a high sensitivity to the target gases.

GasFET: WO_3 layers deposited in-situ at elevated temperatures have been successfully manufactured on GasFET structures by OU-FETF and Picodeon. The in-situ deposited, more compact WO_3 thin films could be used together with sputtered iridium on top. These samples were shown to be functional sensors, and could be tested as a sensing element towards VOC’s. Gas test results showed that the choice of the sensing layer may significantly affect the sensor response. Pure porous WO_3-gate GasFETs showed poor sensitivity and stability, as well as a short lifetime, and are therefore not suitable as gas sensors for the target application. The deposition of porous Ir on top of a dense thin film of WO_3 significantly improved the sensor performance. The sensor response to 100, 50, and 10 ppb C_6H_6 in dry air of an Ir/WO_3 GasFET is significantly higher at the onset of the saturation region (drain-source voltage, V_{DS} = 2.2 V) than in the linear region (V_{DS} = 1.0 V). Ir- and Ir/WO_3-gate GasFETs were operated for several hundred hours without significant changes in response, demonstrating good repeatability. In comparison to pure Ir, Ir/WO_3 showed enhanced selectivity to $C_{10}H_8$.

Process development

SiCFET: Regarding the GasFETs, it has been shown that the standard photolithographic patterning and subsequent lift-off procedure is possible to use for the structured low temperature deposition of at least WO_3. The PLD-deposited Ir and IrO_x GasFET samples have been characterized by SenSiC and found to exhibit the same electrical and gas sensing characteristics as sputter-deposited films. In addition to what was evidenced from the structural analysis performed by OU-FETF, these results also show that the desired structure (on the nano-scale) is obtained with the use of pulsed laser deposition for deposition of the catalytic gate electrode on GasFET sensors. Furthermore, the retained electrical characteristics show that the deposition as such has had no adverse effects on the transducer itself. This flings the door wide open to also process the gate electrode (in addition to the gate passivation layer) with PLD, reducing the number of individual steps in the overall sensor production. Characterization of the electrical and sensor performance (gas sensitivity, stability etc.) for the smooth, homogeneous thin-films of WO_3 deposited by PLD has also been performed, from which it was concluded that the more dense WO_3 films deposited in-situ at elevated temperatures exhibit good sensitivity and stability properties. In conclusion, PLD-deposition of both good quality thin-films of WO_3 at elevated deposition temperatures as well as the catalytic Ir (or IrO_x) gate material has shown that both the gate passivation and gate contact material of GasFET devices can basically be deposited in the same process without breaking the vacuum, which is of clear advantage to device performance.

Samples for process development were provided to Picodeon to carry out picosecond pulsed laser deposition of gas-sensitive layers on FET devices, which were then cleaned after deposition and handed to LiU for gas sensing characterization. Some current leakage problems were encountered for a majority of the devices for which the gas-sensitive layers were deposited by picosecond PLD. SEM-, SIMS-, and C/V-investigations were carried out to understand this problem. What could be concluded is that there are no problems with the deposited films as such, the gate contact films (or any material from these films) do not extend outside the gate area and so cannot make any contact to other electrodes/ terminal. Furthermore, the structure of the films and the surface of the substrate seems to be unaffected. Except for a slightly higher presence of sodium, chlorine, and fluorine in the contacts than commonly found (which might be due to manual handling of the samples) no other deviations were found from the SIMS-characterization. The performed C/V-measurements indicated that the gate insulator suffers from a lowered barrier and, most likely, F/N-type gate leakage. The reason for this barrier-lowering is very difficult to assess, however, since it may have a number of different causes, ranging from local overheating during the RTA process when the basic GasFET devices were processed to a faulty cleaning process prior to gate deposition.

MOS: SnO2 layers that had been PLD deposited by OU-FETF showed to stand very well the post-PLD dicing. This is an excellent result as it should allow for avoiding the pre-PLD-dicing and thus should make the MOS sensing layer deposition by PLD high-volume-production-compatible. Also, the PLD depositions for the photoresist masked SGX platforms were performed by OU-FETF and Picodeon. The results showed that it is possible to pattern also the MOS layers by using lift-off process. Also, the adhesion of the layers to the masked area is excellent.

Scale-up

From the trials described above it is concluded that the full wafer coating will have to be carried out on non-pre-diced wafers and that the dicing will have to be carried out post-PLD. Full-wafer deposition tests at Picodeon provided important information for further development of the deposition process and hardware. Also, successful demonstration of full-wafer deposition capability on dummy wafers (without sensor platforms) both for dense layers suitable for GasFET and porous layers for MOS was performed.

WP3: MOS sensor with nanostructured gas-sensitive layers

Objectives of this WP

To develop MOS gas sensors with so far unachieved performances in terms of sensitivity to low concentrations of the target gases identified in WP1.2 selectivity in presence of interfering gases (also identified in WP1.2) low power consumption and long term stability. To reach this objective, novel micro-hotplates were developed. These new platforms were PLD-coated with optimized layers and the resulting sensor devices were characterized both in lab and field tests (WP10).

Summary of significant results

The fabrication of optimized micro-heater platforms according to SGX's design was completed at two different suppliers. Samples of these optimized micro-heater platforms were post-processed by SGX and supplied to OU-FETF and Picodeon for PLD-coating. Further samples of optimized micro-heater platforms were assembled and electro-thermally characterized by SGX. All the characterization tests carried out by SGX allowed for selecting the best variant out of the 292 tested ones. Additional samples of optimized micro-heater platforms were assembled at SGX and provided to 3S and NanoSense for setting-up the operation and read-out electronics (input to WP6).
Test results of the sensor element characterization and their correlation with material analysis were

discussed between USAAR-LMT, OU-FETF, SGX, and Picodeon. Consecutively, the WP3 partners drew a strategy regarding the choice of PLD layers and defined a new PLD layer trial plan for pre-field tests, the results of which led to the selection of the most promising scenario specific gas sensitive material for the final field tests for the detection of target VOCs (formaldehyde, benzene and naphthalene).
According to the strategy previously defined, OU-FETF deposited four different types of sensing layers on the new sensing platforms provided by SGX. SGX received from OU-FETF this first set of PLD-coated optimized micro-heater platforms, diced these into individual chips, assembled samples of these in to SMD ceramic housings, and provided them to USAAR-LMT for pre-field tests and also to 3S and NanoSense for the field tests. SGX also prepared optimized micro-heater platforms with two of their commercial sensing layers as reference, assembled samples of these into SMD ceramic housings, and provided them to USAAR-LMT, 3S and NanoSense for the field tests. After redefining the ps PLD deposition process, Picodeon also produced sensing layers for final testing.
SGX upgraded their Dynamic Gas Test System (DGTS) to be able to test Benzene as well as lower concentrations of CO, NO2, and Formaldehyde. Then, SGX tested/screened (in constant operating mode) 61 different PLD MOX material families under CO, NO2, formaldehyde, and benzene.
In parallel, USAAR-LMT also tested sensor devices with PLD WO3 sensor layers, produced with both the ns- and the ps-PLD-technology, but these tests were carried out in cycled mode and with ppb-level concentrations of the defined target gases. Based on these results, the PLD layers for further testing and for use in the field tests were selected.
• 292 different variants of optimized Pt micro-heater platforms were obtained from 2 different suppliers and characterized. The temperature uniformity of all of them was good. The passivation quality between heater and sensing electrodes turned out to be good (well insulating) on all but 36 of the 292 variants. Eventually, among the 7 best variants of micro-heater platforms, one was chosen, among other things because of its lower heater resistance level and the resulting working point below 1.8V which is a standard for mobile applications. The chosen design reaches the maximum specified temperature (450°C) at about 1.5V and the cleaning working point (400°C) is reached at about 1.4V. The sensing working point (300°C) is reached at about 1.1V. With the chosen designs, an accelerated lifetime test was performed at 1.6V i.e. slightly above the maximum working point. The test has been running for over 8 months now and shows excellent heater stability with less than 0.3% heater drift over the whole operation time at 1.6V. Thus, with the chosen design the major goals of low power consumption and long-term-high-temperature stability have been achieved.
• All partners involved in WP3 jointly defined the materials to be the best candidates as scenario specific gas sensitive layers for MOS sensors for the detection of naphthalene and for the detection of benzene. Formaldehyde can also be detected with these layers to the much higher threshold limit concentrations.
• The scenario specific layers were deposited by OU-FETF onto the new SGX microheater sensing platforms and delivered to SGX. The layers are formed of nanoparticles with a fractal, tree-like morphology. The valence states of the samples are highly dependent on the used partial pressure when the XPS data is measured after the PLD process done in RT. On the other hand, after the post-annealing processes made in a furnace to the layers, the surface valence states of the materials are almost identical, regardless of the PLD parameters used during deposition.
SGX assembled the first PLD-coated optimized micro-hotplates received from OU-FETF into SMD ceramic housings next to blank or MOF-coated micro-pre-concentrator chips (out of WP5), and provided them to USAAR-LMT for pre-field tests and also to 3S and NanoSense for further integration into the field test systems and testing in the field tests (WP10).

In parallel, Picodeon optimized their USPLD process for producing nanoparticles suitable for doping of sensing layers.
Finally, Picodeon was also able to successfully produce samples with optimized layers using the ps PLD process.
• All sensor types of the third PLD layer generation from OU-FETF and Picodeon were tested by USAAR-LMT for their response to the three main target gases. The sensor layer types were ranked by their overall performance and the four most suitable layers (from OU-FETF) were chosen to be subjected to more thorough pre-field test in combination with pre-concentrators. From these pre-field test results, the best in class PLD layer was chosen to be subjected to the field tests along with an SGX sensor layer.

WP4: SiC GasFET with nanostructured gas-sensitive layers

Objectives of this WP
To develop a SiC field effect based VOC sensor platform with so far unachieved performances in terms of sensitivity to low concentrations of the target gases identified in WP1, selectivity in presence of interfering gases (also identified in WP1), low power consumption and long term stability. To reach this objective, novel GasFET platforms were developed. These new platforms were PLD-coated with optimized PLD gas-sensitive layers and the resulting sensor devices were tested.

Summary of significant results
In two transducer design development iterations SiC-based MISFET-devices with improved thermal time constants, somewhat reduced power consumption, more precise temperature control, and better long-term stability at the highest operating temperatures have been fabricated, facilitating reliable high-temperature processing of high-quality gas-sensitive gate materials by pulsed laser deposition of both metal oxide and metal thin-films as well as reliable high-temperature packaging and operation. The improvements have been realized through further development and adjustments of the chip-integrated heater and temperature sensor structures as well as new designs of the metallic interconnects and passivation layers of the device.
Pulsed laser deposition processes for the growth/ deposition of high-quality insulating metal oxide as well as catalytic metal and metal oxide films as gate passivation and contact materials in SiC based MISFET devices have been developed and applied for the fabrication of highly sensitive MISFET-sensors for VOC detection. The effect of different PLD deposition procedures/ parameters on the materials structure and corresponding gas-sensitivity has thereby been thoroughly evaluated and the deposition conditions/ parameters optimized. A combination of a dense (almost epitaxial) layer of WO_3 and a porous iridium film could in this optimization process be concluded as the most sensitive and selective gate contact design for e.g. formaldehyde monitoring. Analysing the effect of the pulsed laser deposition process on the FET platform has not revealed any significant negative impact on the MISFET transducer, at least for the deposition of thin films.
The optimized WO_3/Ir-gated MISFET sensor devices have been successfully processed through PLD deposition of the gate materials onto the optimized MISFET-platform. The patterning for the deposition may be done through physical masking or photolithographic patterning due to the self-aligned processing.
In order to select gas-sensitive materials/ materials combinations optimized for the defined scenario(s) from WP1, extensive testing of SiC MISFET sensors to determine sensitivity, selectivity, and stability have been performed by LiU, from which it has been shown that reliable responses to single-digit/ few tenths of ppb of e.g. formaldehyde and benzene can be achieved when optimizing the materials design.

From the electrical and gas response characterizations performed earlier in the project on the Silicon carbide Metal Insulator Semiconductor Field Effect Transistor (MISFET) based sensor devices with Pulsed Laser-Deposited (PLD) nanostructured gate contacts (the latter corresponding to the gas-sensitive electrode of the device) developed and fabricated at OU-FETF, the main conclusions were as follows; 1) The insulating properties and stability of semiconducting metal oxides as gate stack materials are impaired with increasing porosity. The usually improved performance of conventional resistive-type metal oxide semiconductor sensors comprised of nano-structured (nano-particles, nano-rods, nano-wires etc.) films does not apply to the Field Effect Transistor based gas sensors. For long-term-stable operation of the MISFET based sensor devices the deposition of a good-quality smooth amorphous/ single-crystalline (with as few grain boundaries as possible, basically an epitaxial layer) and stoichiometric oxide thin-film with as electrically insulating properties as possible is thereby the best option. Such film properties do, however, require the deposition to take place at high temperature and low ambient pressure (commonly referred to as in-situ conditions). 2) The packaging procedure based on direct integration and co-firing of the sensor chip with Low-Temperature Co-fired Ceramics (LTCC), developed during the project (see also WP8) in order to encapsulate the sensor chip and facilitate electrical connectivity to the sensor driving and signal acquisition/ processing circuit board, has some impact on the outer protective passivation layer of the FET-based sensor chip. Involving a high-temperature processing step the packaging procedure might risk to impair the sensor characteristics from damaging this passivation layer.

In order to facilitate both reliability of LTCC-packaged devices, by avoiding formation of micro-cracks in the passivation layer, and reliable in-situ Pulsed Laser Deposition (PLD) for the realization of good-quality smooth and electronically insulating metal oxide thin-films, the design of the basic MISFET transducer has been re-worked. After thorough analysis it could be concluded that the outer passivation layer micro-cracks result from a substantial difference in thermal expansion coefficient (CTE) between the device interconnect metallizations, located in the surface region of the sensor chip just beneath the outer chip protection, and the passivation layer material when subjected to the highest temperatures during the packaging process (based on direct co-firing/ sintering of the LTCC material with the SiC MISFET-chip). Furthermore, from other analyses performed in connection to the Pulsed Laser Deposition of metal oxides as gate material in the MISFET sensor devices it has been concluded that formation of good-quality, smooth and “single-crystalline” films on top of the thermally grown silicon dioxide (SiO_2) require the deposition to be made at temperatures approaching the same temperatures as for the above-mentioned packaging procedure (For further details on Pulsed Laser Deposition of metal oxides, see WP 2), thus most probably could cause the same kind of problems with the protective layer as observed for the packaging procedure.

The problem of micro-crack-formation has been solved by changing the design of the interconnect-metallization. Also the thickness of the diffusion barrier in the outer protective layer was changed to reduce the impact of mechanical stress on this layer. By introducing these changes to the chip design/ layout it was shown possible to subject the device chips to both constant and temperature-cycled heating up to the peak packaging process- and metal oxide deposition-temperatures without the introduction of micro-cracks. The continuous and temperature-cycled heating tests were performed over a total duration of two weeks and thereafter analysed by SEM and surface sensitive EDX to evaluate any evidence for the formation of small cracks in the passivation layer. No evidence for such cracks could be found.

A scheme for processing good-quality metal oxide thin-films through pulsed laser deposition has been developed by OU-FETF (see WP2), e.g. for the fabrication of good-quality WO_3 films. On top of devices for which such good-quality metal oxide gate stack materials were processed, electrically conducting gate electrodes from iridium (Ir) or platinum (Pt) were deposited by DC magnetron sputtering at an elevated background Argon pressure (in order to form nano-crystalline films allowing gas diffusion through the conductive gate electrodes and direct gaseous access to the metal oxide surface underneath), and the devices electrical performance characterized. Additionally, the possibility for conductive gate electrode fabrication using pulsed laser deposition directly has also been investigated by OU-FETF, depositing and characterizing nano-structured films of Pt, Ir, and iridium oxide (IrO_2) on the MISFET platform.

In order to commercialize MISFET sensor devices for which the gas-sensitive and gate passivation materials are processed by pulsed laser deposition (as has been concluded beneficial from a few aspects, ranging from film quality/ stability to sensitivity), it is necessary for the deposition process to be compatible with wafer-level processing. Tests have therefore been conducted in order to develop and transfer the process(es), which were developed by OU-FETF, to a highly automated deposition system which can handle 100 mm wafers.

For evaluation purposes, after the first process-parameter-tuning had been performed, a first round of WO_3 deposition on MISFET-devices was carried out, followed by DC magnetron sputtering of a thin, porous iridium film as electronically conducting gate contact and subsequent electrical and gas sensing characterization. Unfortunately, basically all devices processed for this first evaluation of the work on scaling up the process exhibited (more or less) current leakage problems. Through a more extensive I/V-characterization it could be concluded that the leakage occurs between the gate and drain contacts of the device. Investigating the lateral dimensions, structure and composition of the gate electrode did not reveal any anomalies, the gate electrode had the same extension, structure (on the micro- and nano-levels), and composition as fully functional devices exhibiting no gate leakage.

From capacitance-voltage measurements it could, however, be concluded that the depletion regime of the C/V-characteristics is extended over a much wider range of voltages for devices exhibiting leakage problems compared to devices with normal characteristics. This is an indication of one or both of two possible causes; Either 1) defects of the gate passivation layer (SiO_2) in the vicinity of the silicon carbide/ silicon dioxide interface have been introduced at some stage of the device processing or 2) some impurity element(s), which can act as mobile donor(s) or acceptor(s), has (have) been introduced into the gate passivation layer at some stage of device processing, or both. Both defects (most likely oxygen vacancies) and charged mobile impurity elements can cause the kind of barrier lowering which leads to current leakage through the gate insulator. It is, however, almost impossible afterwards to assess which step in the device processing which is behind the introduction of defects/ impurities, it could basically happen in any of the processes from Rapid Thermal Annealing (RTA) of processed devices to iridium gate contact deposition.

The application of different gate voltages can be quite a powerful tool to enhance the sensitivity towards certain substances over others. With the application of different biases different electronic potential situations at the gate electrode are created, which influences the kinetics of different reactions on the surface of the gate contact, thereby enhancing or decreasing the likelihood of these different reactions to occur. Some reactions/processes (the nature of which presently unknown) in the detection to formaldehyde are influenced by the application of a negative bias. By the application of a negative voltage of 3 V the sensitivity to formaldehyde can thus be enhanced, while the sensitivity to, e.g. benzene remains unchanged.

Thus, various sensor operation modes (gate bias, temperature etc.) allow to obtain the best information on the presence and concentration of different substances. As an additional dimension to the area of sensor operation modes, the operation can be both static and dynamic, in the former case operating each sensor device at a certain constant temperature and a certain fixed gate bias, combining a number of such devices to acquire the information necessary to monitor the VOCs of interest. The other approach, useful for applications with less demands on high temporal sensor signal resolution (as is the case for air quality monitoring), is to operate a single (or possibly a few) sensor device(s) dynamically, cycling the operation temperature and gate bias to sequentially generate more information from that single sensor device. This approach was studied in detail in WP7.

To be able to operate the sensor devices in dynamic mode, the effect of temperature and/ or gate bias cycling on the long-term stability of the transducer/ sensor device has to be assessed to obtain information on possible drift phenomena/ sensor life-time issues occurring when the sensors are operated in dynamic compared to static mode and their causes. Temperature cycling and its effect on sensor performance has been reported earlier. Tests were performed for gate bias cycling of sensor devices over 1.8 • 109 cycles (corresponding to 1000 hrs of continuous operation). It was observed that the transistor characteristics are retained over the 1000 hrs of gate bias cycled operation, though with some slight changes in saturation current over time, attributed to slight changes in leakage current through the device. The changes in saturation and leakage currents were not systematic, however, both increasing and decreasing over time. A more complete I/V-characterization performed after the termination of the gate bias cycling test (after 1000 hrs) strongly indicate the small current leakage to occur over the substrate npn-barrier, and could thus be related to the movement of defects (such as dislocations) inside the SiC epitaxial layers. For the development of VOC sensors based on the MISFET platform, the transistor transducer is combined with gate stack materials which facilitate interaction between the VOCs of interest to monitor and the gate materials such that the electric gate-to-substrate field is modulated. The most promising materials combination among the investigated materials for stable and sensitive detection of at least a couple of the project target substances (benzene, naphthalene, formaldehyde) has been identified as a conductive porous catalytic metal layer on top of a dense, smooth high-quality and insulating WO3 film. This material combination was shown to be extremely sensitive and to show good selectivity to formaldehyde; thus, the catalytic metal/WO3-gate MISFET was chosen to be the scenario specific sensor for formaldehyde in this project.

Pure metal gated (Ir, Pt) as well as metal/metal oxide gated (Ir/WO3, Pt/WO3) GasFET sensor devices have been processed and studied in more detail regarding gas sensing characteristics. The sensors' performance was investigated under static and dynamic operation at ultra-low concentrations. As mentioned above, pulsed laser deposition (PLD) and magnetron sputtering were used as deposition techniques. The sensor performance was studied as a function of the operating temperature from 180 to 400 °C as well as of the electrical operating point of the device, i.e. linear, onset of saturation, and saturation mode. The electrical characterization was carried out on each sensor device before and after gas tests to verify the proper operation of the transistor. High sensitivity to 10 ppb benzene and 5 ppb formaldehyde was demonstrated for Ir/WO3 GasFETs during several repeated measurements. Measurements performed in saturation mode gave a sensor response up to 52 % higher than those in linear mode.

Temperature-cycled operation and multivariate statistics have been used to compare the selectivity of porous iridium on a layer of silicon dioxide (Ir-gate GasFET) or tungsten trioxide (Ir/WO3-gate GasFET), in different mixtures of ethanol and naphthalene. Ir/WO3-gate GasFET sensors show a better selectivity towards naphthalene and can quantify legally relevant concentrations up to 5 ppb with a precision of 2.5 ppb, independent of a changing ethanol background between 0 and 5 ppm (WP7).

Towards the end of the project, Pt/WO3-gate GasFET devices have been processed and tested in static mode operation. Thin films of WO3 were deposited by PLD using 400 or 1000 pulses. The same metal thickness (Pt, 25 nm) was sputtered on top of the two kinds of metal oxide (WO3) thin films. Sensor response to formaldehyde from 25 to 500 ppb was studied between 170 and 308 °C in both kinds of sensor devices. The Pt/WO3 GasFET with 400 pulses performed much better than the one with 1000 pulses. The response at 170 °C was still well-defined in the whole range of concentrations investigated, but very slow.

WP5: µ-pre-concentrator based on nano MIP/MOF layers

Objectives of this WP
• To develop a pre-concentrator device consisting of a micro-heater unit coated with specific nano MIP or MOF layers for the enrichment and concentrated release of relevant VOCs.
• To develop an efficient deposition technique of the pre-concentrator layers on the micro heater platforms for efficient mass production.

Summary of significant results
Different MOFs with and without hydrophobic properties were applied by drop coating and later on by screen printing onto micro-heaters. This method involves an airbrush process of the MOF suspension. MOF UiO-66 was found as the one with the best adsorption properties for benzene and with the lowest influence of humidity. In addition, SGX designed mini-stencils for screen-printing of MOFs by hand on wafer bits as well as a full-wafer stencil for wafer-level spray coating. The MOF-coated samples received from FhG-ICT were evaluated at SGX by optical inspection and thickness measurements. All MOF screen-printing and spray coating results were discussed between SGX and FhG-ICT in order to define possible improvement routes, mainly regarding adhesion and size-control of the printed MOF layers. Subsequently FhG-ICT established a screen-printing method combined with airbrush spraying enabling the coating of 100 micro-heaters in one step. The layer thickness can be adjusted by the number of airbrush cycles in the range of 100 µm so that the adsorption capacity of the selective material is sufficient for the enrichment process. SGX prepared and supplied to FhG-ICT thousands of optimized micro-heater platforms. SGX also completed the characterization of the optimized micro-heater platforms and test measurements with several MOF materials deposited on Al2O3 substrates have been performed by USAAR-LMT using the project target gases, especially benzene.
SGX has characterized the 1st generation of optimized micro-heater platforms for PCs completely. Over 5’000 samples of optimized micro-heater platforms for PCs have been delivered by SGX to FhG-ICT for MOF coating. First MOF screen printing trials were carried out at SGX and the results were used at FhG-ICT for the development of the optimized coating technique. The optimized micro-heater platforms for pre-concentrators showed excellent long term stability.
From the discussions around the pros and cons of the different tested coating techniques between FhG-ICT and SGX, it was concluded that spray coating, thanks to its ease of implementation, was the method of choice for fast development but that screen printing would remain the method of choice for volume production. Mini-stencils (for wafer bits) as well as full-wafer stencils have been designed, fabricated, and successfully used for patterning spray-coated MOF layers on optimized micro-heater platforms. The maximum of the achieved layer thickness is in the range of 100 µm.
Test measurements at USAAR and FhG-ICT with integrated pre-concentrator/gas sensor systems showed good adsorption properties for benzene for all used MOFs, but only UiO-66 and functionalized

HKUST-1 worked also in varying humidity conditions. Due to the best adhesion properties and long term stability all further coatings and field tests were done with UiO-66.

WP6: Sensor system operation and read-out electronics

Objectives of this WP
• To define electronic and associated software to drive sensor and pre-concentrator with parametric temperature cycles and read out sensor data of MOS and SiC GasFET sensors
• To realise hardware modules for operation and read-out of MOS and GasFET sensors as well as operation of pre-concentrators for testing as well as system integration
• To realise a sensor system electronics architecture for multisensor integration and evaluation
• To realise software modules for data evaluation based on temperature cycled operation and algorithms identified in WP7

Summary of significant results
USAAR-LMT: The optimised electronic layout minimizes inductive and capacitive coupling to external noise and enables measurements down to the 100 pA range at scalable sensor voltage. The measurement range is much higher than for other setups especially for electronic modules without logarithmic amplifier. The standardized signal to noise ratio (SNR) is the SNR for a relative resistance change of 1%. Various realisations of the sensor read-out electronic Log112/114 are all gaining a high signal to noise ratio up to a resistance of 10 GΩ.
SGX: Electrical characteristics of the 2 new generations of micro-hotplates for MOS sensors measured in the frame of WP3.1 were analysed and compiled, and, from there, the curves of T° vs. Rh, Vh, and Ph were drawn and used as input for the fine-tuning of the operation & read-out electronics. Electrical characteristics of the new generation of micro-hotplates for micro-preconcentrators measured in the frame of WP5.2 were analysed and compiled, and, from there, the curves of T° vs. Rh, Vh, and Ph were drawn and used as input for the fine-tuning of the operation & read-out electronics.
3S: A small series of field test demonstrators has been produced for both MOS (20 devices) and GasFET (10 devices) sensors and provided to USAAR-LMT as well as LiU for field evaluation.
SenSiC: From 3-months of continuous operation of packaged sensor devices, stable operation of the heater resistor and the temperature sensor in packaged sensor elements could be concluded over the whole range of intended operating temperatures.
NanoSense: A small series of field test demonstrators has been produced with SGX sensors and Figaro TGS8100 for comparison and without pre-concentrator.
Thus, in this work package, different electronics modules were developed and tested for the two different sensor principles studied in the project as well as the novel pre-concentrator concept. The modules were thoroughly tested and provided the basis for the system integration in WP8 as well as the implementation of the dynamic operation modes and the integrated data processing developed in WP7.

WP7: System operating mode and data processing

Objectives of this WP
• To ensure the required sensitivity for the defined target gases and the required selectivity in the defined operating scenarios vs. interfering gases

• To provide feedback information and specifications for material deposition and device integration according to the requirements of system operating mode and data processing
• To develop the necessary algorithms for data evaluation, suitable for integration in the sensor system electronics
• To define the necessary parameters to be determined by calibration of individual sensor elements
• To define the necessary parameters to be determined during sensor system calibration

Summary of significant results
OU-FETF carried out long-term measurements of the MOS sensing layers, and SGX completed stability tests of the micro-heater platforms for the MOS sensor. In addition to the original work plan many tests have been done in a sensor pre-concentrator arrangement to consider the interde¬pen¬dence of the sensor operation for pulsed gas concentration (pre-concentrator emission) at an early stage of development. USAAR-LMT developed and tested temperature cycles for the optimized, joint operation of MOS sensor and pre-concentrator to increase sensitivity and selectivity towards the target gases. 3S has discussed the MOS sensor operation with the relevant partners to find specifications for hardware/firmware, also considering the influence of data evaluation on read-out.
OU-FETF carried out long-term measurements of the GasFET sensing layers. LiU has shown that the required sensitivity (low ppb-level) can be reached for the target gases with optimized sensing layers. USAAR-LMT developed and tested temperature and gate bias cycles to increase selectivity towards the target gases, with a focus on a new sensing layer out of porous iridium on a dense WO3 layer. 3S has discussed operation of the GasFET sensor with the relevant partners to find specification for hardware/firmware and software setup and adaption, including new specifications for read-out.
FhG-ICT prepared samples of optimized MOF layers on micro-heaters for gas tests at USAAR-LMT and stability tests at SGX. Temperature limits for stable operation modes were defined. 3S discussed pre-concentrator operation and circuit implementations (cut-off circuit) with USAAR-LMT. An adequate firmware implementation has been fixated. NanoSense tested the preconcentrator temperature control by PWM in order to improve electronic efficiency and reduce BOM.
USAAR-LMT tested both MOS and GasFET systems with application specific, complex gas tests, and optimized the system operation mode based on the evaluation results. Evaluation strategies for best sensitivity and selectivity were developed. Successful quantification of naphthalene in changing ethanol background was possible, and a strategy for the combination of cycles with unequal length was devised. LiU contributed with static tests and evaluation of GasFETs with optimized sensing layer in various conditions. 3S implemented, tested and updated the agreed-on operating modes.
Naphthalene in the ppb-range can be quantified in changing ethanol background. The newest generation of GasFETs with porous iridium on a dense WO3 layer exhibits a rather binary response to ethanol. That means that a large signal change is seen between no ethanol at all and presence of some ethanol, however, the actual concentration has little influence. Porous iridium on SiO2, instead, produces distinct signal changes with each change of ethanol concentration. Consequently, GasFETs with WO3 layer are less influenced by changing ethanol background concentrations, enabling considerably better quantification which was tested for naphthalene compared to porous Ir without WO3. The tested naphthalene concentrations are between 0 and 40 ppb, quantified independently of the presence of 0, 1, 2.5 or 5 ppm ethanol.
The best strategy for combination of cycles with unequal length has been determined. Microstructured MOS sensors have a significantly lower thermal mass than GasFETs, leading to different appropriate

lengths of their respective temperature cycles, i.e. longer cycles for the GasFET. Data simulations have been conducted to find out how to best combine those two different cycles to achieve the best overall result. It has been shown that computing features from the long cycle as well as from the mean of all short cycles within the same time interval achieves best performance.

For the integrated pre-concentrator / MOS gas sensor system, a combined operating mode has been developed. The temperature cycles of both devices must be coordinated and synchronized to achieve the best gas sensing performance. A combined temperature cycle with a length of 120 s was used for testing. In this cycle, the gas sensor temperature is increased from 200 °C to 400 °C during pre-concentrator desorption, and then the temperature is kept constant and decreased again before the PC is switched off. The same sub-cycle is repeated during gas re-adsorption at the pre-concentrator.

MOS and GasFET systems have been tested with complex gas profiles (two humidities, four VOC background variations, several concentrations of all target gases; refer also to WP10). To evaluate sensor performance, a model was trained to discriminate low from high formaldehyde concentrations, independent of all other gases. This model performed poorly because it mainly optimizes the abundant “0” class and ignores the scarce “1” class. Equalizing the size of both classes improves the performance significantly and enables the model to detect too high formaldehyde concentrations in 80 % of all cases, as opposed to 30 % with the initial training approach. The trained model can predict the whole dataset with very similar accuracy. The data were taken from a TCO-GasFET with new sensing layer; however, the results are also applicable for MOS sensors and other forms of cycled operation.

These results can be improved further by using a hierarchical classification approach. In this approach, both classes (“0” and “1”) are correctly predicted in approx. 90 % of the cases despite varying interferent concentrations. This approach requires somewhat more computation time than the non-hierarchical approach; however, evaluation is still quick with the chosen model type even on low-cost hardware. The more likely bottleneck is the large amount and variety of training data required to cover all possible cases with sufficient resolution. Thus, the hierarchical approach should only be considered in sensitive environments where the benefits outweigh the costs, e.g. hospitals or kindergartens. The data used for development of the presented strategies stem from a GasFET with the new sensing layer operated at five different temperatures in each cycle. However, they can be applied to MOS sensors for significant classification performance improvements as well.

Both sensor types can discriminate different levels of relative humidity (r.h.) very well even in the presence of interferents. With the dedicated, built-in humidity sensor, this information potentially enables self-monitoring of the systems and early detection of failure.

Several different hot plate designs were successfully coated by FhG-ICT with MOF layers and provided for gas adsorption measurements and stability tests at USAAR and SGX. These results were used as a basis to specify valuable parameters and limits for the operating mode.

The sensing layers on top of the MOS sensor have been studied by OU-FETF group by electrical gas measurement at 350 °C up to 100 hours. Only a small drift was noticed on the sensor signal. Only negligible growth in grain size was noticed in the sensing layers after the different post-annealing processes in a furnace, e.g. at 450 °C for 3 hours and 400 °C for 12 hours. The XPS studies of the sensing layers revealed that the post-annealing processes have a clear effect to the sensing layer surface valence states. The nanotree layers showed almost identical XPS curves after the heat treatments regardless of the used PLD parameters. The studies made to the MOS sensing layers returned to OU-FETF after measurements at USAAR revealed a carbon contamination surrounding the membrane of the microheater. The effect of this contamination to the sensing performance is still unknown.

In tests at LiU, pure Ir-gate GasFETs show higher sensitivity to the three target gases (i.e. formaldehyde: 0.2 ppb in dry air, 10 ppb under 60% r.h.; benzene: 0.5 ppb in dry air, 1-3 ppb under 60% r.h.; naphthalene: below 0.5 ppb at any studied humidity level up to 60% r.h.). However, Ir/WO3 GasFETs show enhanced selectivity to naphthalene. The relative response to formaldehyde and benzene with an Ir- and an Ir/WO3-gated GasFETs operating at 300 °C was compared. Good stability and repeatability of the sensors were demonstrated by 1) operating the sensors for several hundred hours at a constant temperature; 2) repeating the same measurement several times; 3) repeating the same measurement after 12+ months under the same conditions.
As mentioned above, repeated tests performed with an Ir/WO3 GasFET and using benzene as target gas demonstrated that the sensor response in saturation region is significantly higher than that in linear region, i.e. 45 % higher at 10 and 50 ppb, and 52 % higher at 100 ppb. The sensor response varied from 6 to 8 % in average when the GasFET was operated at the saturation region or at the onset of saturation.
In long-term tests at SGX the excellent stability of the optimized micro-heater-platforms for MOS sensors was shown, with a heater resistance drift of less than 0.3% over more than 8 months of operation at operation temperatures above 450°C.
TCO control implementation was completed by 3S and adapted according to the findings at USAAR-LMT to allow specific benzene detection in stand-alone operation.
NanoSense validated a high efficiency and low-cost, low BOM pre-concentrator PT heater driving method. Furthermore, a high-speed fuzzy logic PID temperature control was validated on PT micro platform. Better temperature stability was achieved for TCO with temperature step changes.
Thus, in this work package, different operation modes were tested with different optimization targets, i.e. gas identification or quantification of a specific target gas. Different data processing strategies were identified for evaluation of sensor systems with single or multiple sensors and for data fusion of sensor with different time scales due to different measurement principles. These results provide the basis for optimized application-specific operating modes, not only for selective detection of VOCs but also other applications.

WP8: Sensor system integration

Objectives of this WP
• To combine nano-sensors, pre-concentrator(s) and electronics for dynamic operation and advanced signal evaluation.
• To develop of suitable housings or arrangements to achieve an optimum efficiency of the pre-concentrator and, if possible, constant gas concentration during one temperature cycle
• To develop a packaging concept and study of the mass production capability for sensor sub-groups and complete sensor systems
• To provide an integrated and functional consistent prototype version of the sensor system
• To evaluate of sensor sub-groups and systems in lab tests with specialized test set-ups to allow reliable detection of very low VOC concentrations in changing background gas compositions under varying ambient conditions

Summary of significant results
USAAR-LMT performed the simulation and assessment of mechanical design of sensor sub-groups for optimal pre-concentrator integration. The arrangement of the pre-concentrator in the system was optimized in order to improve the quality of the FEM simulation and to predict the impact of the operation

mode. This clearly improved the development of operation modes for the pre-concentrator and the integrated system.
The packaging concept for “MOS sensor + micro-preconcentrator” sub-groups was developed by SGX according to the results and conclusions from the simulations. This concept combines one micro-preconcentrator chip with 1 or 2 MOS sensor chips side by side in a standard and very compact ceramic SMD package with a dedicated metal cap also designed according to the conclusions from simulations to achieve the desired performance.
Hundreds of sensor sub-groups according to the concept developed in simulations were produced in SGX’s production line with SGX’s standard production equipment. This allowed for assessing the production capability for sensor sub-groups and for verifying that this production process is high-volume-compatible and low cost.
LiU contributed to identify suitable integration strategies, compact and space-saving housing of the SiC-FET sensor system, which were designed and realized. Close collaboration with SenSiC allowed development of a versatile and cost-efficient packaging based on low-temperature co-fired ceramics, LTCC. SENSiC succeeded in the LTCC based direct packaging of GasFET devices which has been qualified with good results.
3S and NanoSense developed and manufactured integrated sensor systems with different electronic approaches. The number of MOS sensor systems produced was limited by the number of sensors available with both satisfactory Pt heater and pre-concentrator. For MOS sensors, the system was designed to fit into a flush mounted casing. SiCFET sensors could not be mechanically and electrically integrated with the MOS sensor sub-systems, therefore 3S developed and produced a specialized device somewhat larger than the modules for flush mounted casing for field-testing of SiC-FET-based sensor systems.
A sensor system was set up and tested for benzene tests at JRC (Joint Research Centre, European Commission). This test system containing three gas sensors has been set up, calibrated and installed at JRC for long-term benzene tests. It did not contain a preconcentrator as these were not available at the time the test systems needed to be delivered. An on-line benzene calculation using PLSR was implemented into the software. In calibration measurements, benzene was applied in concentrations from 0.5 to 10 ppb, with varying gas humidities and interferent gases. The PLSR achieved for simple conditions (zero air as background, one humidity (25%RH) and one low concentration interferent gas, here 2 ppb of toluene) a quantification uncertainty well below 1 ppb. By increasing the complexity of the gas mixture, the reliability of the quantification drops: a PLSR for benzene in a more complex background variation (two gas humidities, 25%RH and 40%RH, background air containing methane, hydrogen and CO as ubiquitous air constituents as well as varied toluene and CO concentrations) still achieves an uncertainty of approx. 1.5 ppb, which is well beyond anything previously achieved with MOS-based sensor systems. The developed PLSR calibration data was implemented in the software for on-line signal processing and benzene quantification.
Low cost, reliable, hermetic packaging of SiC FET based sensors has been realized through direct integration of SiC sensor chips in an LTCC package. The chip is added through pick-and-place procedure to the LTCC package prior to lamination and sintering (ramping to 865 °C, 30 mins at peak temperature), thus automatically integrated in the package (essentially becoming part of the package itself) and abolishing any needs for die attachment (gluing, soldering), (wire) bonding, and sealing of the encapsulation. The packaged device is solderable to e.g. standard printed circuit boards. Flip-chip bonding to a circuit board is also possible. As previously reported, electrical testing of packaged devices

have shown that normal transistor Ids/Vds-characteristics are retained for packaged sensor devices, though with a slight change in threshold voltage. No evidence for any increased leakage across the body diode or the gate dielectrics has been detected. The gas sensing performance has also been shown to be retained.

From the long-term testing no significant effect of the packaging on gas response, sensitivity, response time etc. has been observed. No evidence for long-term problems regarding the structural/ compositional integrity of the packaging or packaged devices have been found, as investigated by SEM, EDX, XPS etc. No differences in the structure or compositions of layers and interfaces between as-processed and 3 months aged devices have been found. LiU contributed to the development of a new packaging concept based on low-temperature co-fired ceramics, LTCC. Other existing networks and collaborations with external partners involved in the development of such LTCC packaging were exploited and with beneficial results for the SENSIndoor project (output to WP11).

The previously used 2D COMSOL simulation model was replaced by a 3D model; the simulated MOF layer thickness and adsorption/desorption parameters were not changed from the original model. The results regarding the gas concentrations at the position of the gas sensors during desorption deviate from the 2D model, the calculated concentrations are lower by a roughly factor of two due to the gas spreading also laterally. The influence of the adsorption time was investigated as well. The results show that an adsorption time of 220 s almost reaches saturation of the pre-concentrator in the adsorption phase and therefore, the maximum concentration in the desorption phase. Longer adsorption times are not necessary for benzene allowing a measurement duration of less than 5 min total. The last generation of integrated PC/MOS systems was tested with benzene and toluene to assess the performance of the pre-concentrator for these gases. These systems use UiO-66 as pre-concentrator.

Different adsorption times for the pre-concentrator were tested. The gas sensor was operated at a constant 1 min cycle (15 s high temperature, 45 s low temperature). The low temperature of the sensor was constant at 250 °C to allow the observation of the desorbing gas using the analysis of the relaxation of the sensor surface after a temperature step. In this operation mode, the slope of the conductance (over time) is in good approximation linear to the gas concentration. This operation mode obviously does not show the highest sensitivity to benzene due to the relatively low temperature and is also different from the operation mode with the best selectivity which uses a temperature ramping in the desorption phase. This mode of operation, however, can serve for a better understanding of the desorption process. The pre-concentrator was not desorbed in every sensor cycle, between one and six cycles were left out to vary the adsorption time.

The sensor under this operation mode is much more sensitive (5-10 times) to toluene. The intensity of the desorption is depending on the adsorption time. For benzene, adsorption times longer than 165 s (more than three periods) do not affect the desorption signal. The sensor is in equilibrium after this adsorption time, which is in good agreement with the simulations. For toluene even longer adsorption times (e.g. 225 s) do affect the desorbed amount. This is presumably due to the lower volatility of toluene. In general, the pre-concentration effect is very small for the quasi-static signal. However, the differential signal during the adsorption is quite significant. The chosen sensor shows a significant difference in gas responses to benzene and toluene. For toluene, the difference between a signal without PC desorption to the signals with PC desorption is much higher compared to benzene. A second sensor with different layer showed similar qualitative response behaviour to the two gases, but the response was in general significantly lower. As already seen in the simulations, it is not necessary to use very long adsorption times for the pre-concentrator. Adsorption periods longer than 3 cycles, corresponding to three min., do not increase the

signal significantly.
To further examine the different PC and sensor performance regarding the two gases, the signal with PC desorption was pointwise divided by the signal without desorption, which generates normalized signals which show the influence of the pre-concentrator. The raw signals for toluene show a much clearer response to the gas compared to benzene. Pre-concentrator desorption also differs, as the difference is more obvious for toluene. The calculated normalized signals confirm this, the benzene signal increases by approx. 35% during desorption, for toluene it is 70%. Furthermore, desorption of toluene happens quicker compared to benzene. The benzene signal was analysed further by looking at the derivative dSig/dt of the sensor signal. The results show that the PC desorption has a clear effect on the sensor signal, as the gradient of the signal is affected by the desorbed gas concentration. This signal slope might also be used as a signal for determining the gas concentration. The advantage of this feature is that it should not be affected by sensor drift and permanent gases, like carbon monoxide and hydrogen that do not adsorb at the pre-concentrator. Therefore, it should be more stable and reliable compared to gas sensor conductance signals, additionally to other features extracted from TCO operation of the gas sensor. For further signal processing, an LDA was performed with the goal to separate the different gases and gas concentrations. For this, the two sensor layers were fused by combining signal features from both sensors. As expected from the raw signals, the LDA yielded better results for toluene for which 98% of all cycles were classified in the correct concentration group. For benzene the rate of correct classifications was still 90%, mostly due to misclassifications of the lowest concentration.
Thus, in this work package, different operation modes were tested with different optimization targets, i.e. gas identification or quantification of a specific target gas. The studies achieved a greatly improved understanding of the performance potential of the novel µ-pre-concentrator approach developed within this project allowing the realization of application specific sensor modules and sub-systems for various applications beyond Indoor Air Quality control.

WP9: Efficient sensor system calibration

Objectives of this WP
• To recommend and provide (proven) cost-efficient calibration processes through the manufacturing process of the sensor system (elements, overall system)
• To recommend calibration intervals (mainly based on the overall system stability)
• To develop a strategy for possible in-application re-calibration during sensor system life-time

Summary of significant results
SenSiC:
From the earlier evaluation of wafer level electrical characteristics the selected parameters for possible inclusion in an in-production calibration scheme - Ids @ specified Vds, Vgs and Vsubs, Ids/Vgs @ specified Vds and Vsubs as well as Isub @ specified Vsubd – have been reviewed in connection to on-line calibration characteristics for sensor units already operated in other running applications, such as real-time flue gas monitoring for combustion control purposes. From the comparison between on wafer tests and the electrical and gas sensing characteristics as well as on-line calibration needs/ measures of individual sensor devices when operated over a longer period of of time it seems to be necessary to complement any in-production calibration scheme with also at least a zero-point calibration after some time of operation in the field. The baseline and response stability after some time of operation is, however, such that repeated

calibration might not be necessary after that.
A wafer-level screening process for selection and grouping of GasFET sensor devices according to fulfilment of minimum requirements and expected performance has been developed based on GasFET I/V-characterization. This grouping might facilitate a kind of in-production calibration of GasFET based sensor systems for the future.
USAAR-LMT:
• Traceability of the gas mixing system was successfully shown.
• A more realistic composition of the calibration was achieved by literature studies
• Integrated MOS sensor systems (3S, NanoSense) and GasFET systems (SenSiC, 3S) have been successfully calibrated
• A traceable field calibration, proven to be comparable to the regular gas mixing approach, was successfully developed and tested
• A zero VOC sample and samples with various predicted concentrations (10 ppb to 10 ppm toluene) have been prepared and tested
• Results have already been published at two conferences and in a peer reviewed publication (C. Schultealbert et al., Sens. & Act. B. 239, 390–396, 2017). The demonstrator was shown at the SENSOR + TEST 2016 fair, May 10th-12th 2016
• The gas mixing system was improved based on the results of a certified test lab for better accuracy and repeatability
SGX:
• Wafer-level testing of heater characteristics is do-able and makes sense but not wafer-level calibration.
• Dispersion measured on heater characteristics eliminates Calibration Approach A.
• Analysis of available facilities and reasonably possible upgrades of these tend to eliminate Calibration Approaches B and C in favour of Calibration Approach D.
NanoSense:
• Individual calibration procedure and method for platinum heater dispersion.
• Mass production low cost individual calibration method and tooling definition for up to 15 000 sensor systems at a time.
• Definition of a special Modbus communication protocol for calibration (orders broadcast + individual data collection). Normally a Modbus is limited to 254 addresses but as calibration of up to 15 000 sensor systems was desired, a 3 layers sub addressing system was implemented.
• Design of a sub calibration board with MCU able to handle 9 sensor systems to be plugged on existing 40 sockets main calibration board.
• Digitalisation of the existing 2CBM calibration room to handle sub calibration boards and collect individual sensor systems data.
3S:
• Strategy for higher volume calibration based on parallel sensor board calibration
• Strategy for initial on-site calibration using specific pre-loading of µPC combined with an on-site start-up-cycle

WP10: Evaluation and testing

Objectives of this WP
• To verify the SENSIndoor technology by testing complex multisensor systems under controlled lab

conditions in complex test scenarios
• To test system-to-system variation in correlation with different calibration schemes
• To demonstrate the functionality of complex SENSIndoor multisensor systems under realistic working conditions and to verify the performance with reference measurements for target VOCs

Summary of significant results
An efficient protocol for lab and field test has been agreed between the partners. The protocol includes time tables for tests and reference measurements as well as operation modes derived from WP7. The aim of the test protocol is to allow a measurement of the system to all target gases in a reproducible reference atmosphere that should represent indoor background air as good as possible. The protocol represents a compromise between the comprehensiveness and the complexity of the testing.
The results of the lab test have been summarized in deliverable D10.2. Several sensor systems have been tested in the lab under application near conditions. This includes the four systems in different configurations (sensors and pre-concentrators)
• 3S µPC / MOS gas sensor systems (17 pcs).
• NanoSense µPC/MOS gas sensor systems (10 pcs).
• SenSiC-3S GasFET systems (5 pcs).
• USAAR-LMT adaptable µPC/MOS gas sensor systems as reference (3 pcs).
The calibration systems (WP9) have been successfully demonstrated to be capable of testing a multitude of sensor systems (WP6) in a very effective way. A large amount of testing data has been obtained to demonstrate the properties of the integrated systems and to identify further optimization strategies for the PUDF (WP11). To this end, various methods of data processing (WP7) have been tested to identify a method that should be used in field tests.
The lab evaluation/calibration measurements include a more complex model of the gas background than most of the previous tests. The need for the adaption of the gas background model was derived from the inter-laboratory calibration trial with the JRC. The lab evaluations show that the increased gas background (permanent gases / VOCs) is a great challenge for the gas sensing performance of the developed gas sensor systems. In these complex environments, detecting and quantifying the target gases in their respective concentration ranges requires a complex signal processing and data evaluation scheme.
All sensors could discriminate the relative humidity of the gas mixture with very high accuracy. This data can be complemented with the humidity signal of the humidity sensor. This information redundancy from two physical sensors can potentially be used for the detection of malfunction or drift within the sensor arrays (self-monitoring). Moreover, the signal can be categorized in different humidity regions in order to improve the quantification of the target gases.
For the target gases various data processing schemes were tested. The simplest approach, the two class problem that would detect any toxic target gas at any concentration above threshold with respect to any other gas composition was proven to be inadequate leading to poor results. These results are not unexpected as the chemical properties and concentrations ranges of the target gases are quite different. A four class problem (3 classes for the target gases above threshold vs. all other gas compositions) is already improving the detection capability. The results can be improved further by using a hierarchical classification approach. The detection of a single target gas above threshold vs. all other compositions already provides a suitable detection mechanism, but it requires three different two-class discriminators. With these methods only a few misclassified incidents are observed. A three class model for the concentration range (0: far under threshold, 1: above threshold and 2: close to threshold but below) can

increase the confidence of the detection. The categorization into the humidity levels prior to the classification was found to be a further improvement. Also, it was shown that sensor fusion of different gas sensing layer materials in the data processing is a promising method to significantly increase the quality of classification results.

However, the results of the adaptable reference system are in general better than the results of the integrated systems. Thus, an optimization of the field test systems seems promising. To this end, we tried to identify the strategies for optimization. The comparative analysis of the signal quality reveals that this is most certainly not due to sensor signal quality caused by different electronic modules. Therefore, aspects of gas diffusion into the sensor module and the thickness of the PC layer seem to be relevant factors. The consumption of gas by the sensor has been underestimated in the prior investigation and should be part of the optimization process.

The sensors for the MOS field test systems have been supplied by SGX. The production at SGX has been planned and carried out according to the requirements defined in pre-field tests. The GasFET sensors were supplied and integrated by SenSiC using an electronic board supplied by 3S. The sensor sub-systems available in the test (two MOS-PC types and one GasFET type) could be assessed in the laboratory and subsequent field tests with hardware systems supplied by 3S. In addition to the planned scope from the DoW, the MOS sensor sub-system could be tested successfully in a specialized benzene detection experiment at JRC, Ispra.

The field test measurement results are summarized in D10.3. For the MOS systems the basic capabilities of the developed gas sensor systems to detect changes of the gas environment in real life conditions could be demonstrated. Transfer from a laboratory calibration to a real world application needs to be studied further. The transfer does not work if the gas conditions in the application deviate too much from the calibration conditions. Another problem might be drift effects in the used sensor systems. This approach can yield realistic results in some scenarios, but should in general be supplemented by in-field calibration of the sensor systems, e.g. with a reference measurement, as was used in these studies.

The GasFET system has been installed in a classroom of the Montessori school Trilobiten in Linköping, Sweden, during June-September 2016. The same sensor system was moved to a private apartment in Linköping, which was newly renovated, during October-December 2016. The sensor system performances were tested and evaluated at LiU before installation in each of the test sites. The operation of the sensor system (temperature cycled and gate bias cycled operation, signal processing, algorithm) was optimized in close collaboration with USAAR-LMT for selective detection of formaldehyde in the low ppb concentration range. A Formaldehyde Multimode Monitor from Graywolf was used as reference sensor for monitoring formaldehyde. This instrument can acquire a measurement every 30 minutes. The response time of the GasFET sensor is <1 minute. A commercial sensor from SenseAir, tSense Touch Screen CO2 + RH/T Transmitter, was also used for continuous monitoring of temperature, humidity level and CO2 concentrations. The sensor signals were remotely accessible via an Intel stick computer with 4G modem which was used for sensor control and read-out.

The concentration of formaldehyde tends to increase in the school overnight (when the ventilation system is off), but does not exceed 35 ppb, whereas it is <10 ppb during daytime, so in all cases below the required threshold. Even for these low concentrations, the evaluation results show good agreement between the reference instrument and the SENSIndoor sensor system. The preliminary results from the field tests in Trilobiten were shown at the Final SENSIndoor Meeting at the presence of the advisory board and during a public session with external partners and general public. These results were also presented at other three international scientific meetings, i.e. the FunMat Final Conference in Linköping (oral), the

MRS Fall Meeting in Boston (invited), and the NACSIC II Workshop at Linköping University (invited).
The sensor with a tailored and PLD-deposited WO3/Ir-gate and run in a temperature-cycled mode according to a scheme developed by LiU and USAAR-LMT was shown to give some promising results regarding the possibility to estimate the variation in formaldehyde concentration in a public school (at levels below the legal limit), even without the use of a pre-concentrator.
Several sensor systems have been characterized at BAM ("Bundesanstalt für Materialforschung und-prüfung") in Berlin, Germany, including two GasFET sensors with porous iridium on either dense WO3 or dense SiO2, respectively, and two MOS sensor systems combining each a MOS sensor and a pre-concentrator. In order to prevent sensors downstream being influenced by the reaction products of sensors upstream, a parallel setup of all six systems was selected. To achieve roughly equal flow rates in all branches, 10 cm of 1/16" tubing was inserted in each branch. This leads to a well-defined and equal flow resistance for all branches, canceling out the influence of differently built measurement chambers.
The same system was characterized at USAAR-LMT with several concentrations of target gases in two different relative humidities (30 and 50 %), as well as four different background VOC cocktails. In a first evaluation of the data, models were trained to discriminate between high and low levels of each target gas separately, independent of humidity, VOC background and the concentration of other target gases. Thresholds were 1.5 ppb for benzene, 2.0 ppb for naphthalene, and 80 ppb for formaldehyde. Everything below this threshold is classified as "0", everything equal or above as "1". The models are trained with equally sized classes and validated with 10-fold cross-validation. Five features are extracted from each GasFET cycle (the mean value of each temperature plateau), and 24 from each MOS cycle (mean and slope of plateaus and ramps).
Neither of the sensors can reach an error below 30 % for naphthalene or formaldehyde. However, combining both sensor technologies ((1)+(3)), the error can be reduced about 10 % in both cases. For naphthalene, addition of GasFET (2) with another sensitive layer brings the error even close to 20 % despite varying interferent concentrations throughout the measurement. Low and high values of benzene cannot be discriminated by either sensor or by any combination of sensors. However, applying a hierarchical model (see WP7) that first discriminates humidity, then VOC background concentration, and finally trains specialized models for benzene detection, the error can be lowered to around 30 % with a combination of (1)+(3)+(4).
In total, the tests demonstrated the potential of the developed sensor technologies to detect and quantify hazardous target VOCs even in very complex background mixtures. It should be noted, that this performance today cannot be achieved with any other sensor technology but would require sophisticated analytical tools like PTR-MS (at cost > 100.000 €) for online detection and quantification. Thus, the outcomes from WP10 were highly effective input to WP11.

WP11: Dissemination and exploitation

Objectives of this WP
• To disseminate the accomplishments of SENSIndoor to target groups, new users and communities
• To exploit the project results and to guarantee their sustainability
• To exchange information and establish relationships with current projects and initiatives
• To ensure that the achievements of the project results are communicated to targeted potential clients and thus in additional market segments

Summary of significant results
The project communication and dissemination systems (project website/intranet, dissemination toolkit, etc.) set-up at the start of the project were widely used among the consortium, creating visibility and project outreach. The website (http://www.sensindoor.lmt.uni-saarland.de/) was continuously updated with project-related news items, relevant conferences and publications, which can be accessed via a web-link under the Publications section (http://www.sensindoor.lmt.uni-saarland.de/publications/index.html).
The SENSIndoor partners produced two newsletters in the course of the project, one near the end of year two and the other towards the end of the project. The e-newsletters are accessible via the website (http://www.sensindoor.lmt.uni-saarland.de/news/newsletter/index.html) and were shared with the SENSIndoor network via e-mail. Hard copies of the newsletter were distributed at various occasions and events. A video was produced which especially demonstrates the novel integrated microsensor concept comprising the pre-concentrator and gas sensor in a single miniaturized package. This video is also available via the SENSIndoor website and via Youtube.
The SENSIndoor dissemination strategy, the IPR management strategy as well as the expected exploitable projects outputs have been outlined in the Plan for the Use and Dissemination of Foreground (PUDF), submitted as preliminary version in month 20 and as final version in month 36. Market opportunities and limitations of the SENSIndoor technologies in relevant market segments have been assessed in the business plan, which was submitted as D11.7.
Interfacing activities promoted the exchange of information with current projects and created opportunities to disseminate the accomplishments of SENSIndoor and raise awareness for IAQ in the general public as well as for interested stakeholders. The cooperation with the SENSIndoor partner projects IAQ Sense and MSP led to the planning of joint presentations and sessions at the Sensor+Test fair in Nuremberg and the Indoor Air conference in Ghent. Furthermore, the project partners actively engaged with related projects and networks, especially the COST action EuNetAir with its workshops and the European Sensor Systems Cluster (ESSC) with two working groups chaired by SENSIndoor project members.
A number of the SENSIndoor partners presented research done in SENSIndoor at various conferences and workshops; a total of 113 dissemination activities were recorded during the project. A total of 17 peer-reviewed publications and 12 papers in conference proceedings and one thesis have resulted from within the SENSIndoor consortium so far. In addition, SENSIndoor results will be presented at conferences, workshops, and meetings in the upcoming months (e.g. partners from LiU and USAAR have been invited to present SENSIndoor results obtained with SiC-FETs at the next Key-VOCs workshop to be held at JRP Ispra, Italy, on 24 March 2017).
The aim to disseminate the obtained results through a workshop at a highly relevant conference in the field of IAQ was achieved by successfully organizing a special session at the 15th EUROSENSORS conference held in Budapest from 04 to 07 September 2016. Accomplishments of SENSIndoor have been disseminated actively to the national and international scientific communities and the general public via the project website, conferences, workshops and publications.
Collaborations with other projects and initiatives have been initiated very early in the project, promoting the exchange of knowledge and synergies between different initiatives. Specifically, the cooperation with IAQSense and MSP resulted in a joint booth and a joint session at the Sensor+Test fair 2016 and a joint presentation with IAQSense at the Indoor Air 2016. The European Sensor Systems Cluster (ESSC) with the working groups focused on Indoor Air Quality (chaired by Prof. Andreas Schütze, USAAR-LMT; COO) and on integration and commercialization (chaired by Olivier Martimort, NanoSense) provided an important channel to raise awareness for IAQ in the general public and interested stakeholders and was

used by partners to further broaden their network.
In the Plan for the Use and Dissemination of Foreground (PUDF), the consortium has outlined strategies to address stakeholders both within and outside the international sensor-community, how to manage IPR and drafted partner’s exploitation plans. Complementary to the PUDF, a business plan has been developed to assess exploitation opportunities in various market segments based on the performance and costs of the SENSIndoor technologies.
The active dissemination policy of the project consortium has led to a range of contacts with potential partners for further exploitation, both in the areas of indoor air quality for various application scenarios but also other fields of application. These contacts are currently pursued on a confidential level by individual project partners.

Potential Impact:
Potential Impact:

As evidenced by the results achieved, the project has addressed a highly relevant application scenario with very high requirements on the sensor performance, mainly in terms of sensitivity (ppb and eventually sub-ppb detection limits) and selectivity (target VOCs against a complex and changing background). These requirements cannot be met by conventional sensors today due to their lack of sensitivity (at least one, often two orders of magnitude too low) and selectivity. While the selectivity can be improved by dynamic operation already today, conventional sensors show a large spread in their characteristics resulting from not well enough defined manufacturing processes for the gas sensitive layer. Due to this spread, extensive calibration of each individual sensor is today required for systems based on dynamic operation, making this approach viable only for niche applications allowing high cost per sensor node. With the technologies and systems developed SENSIndoor has demonstrated that nanotechnology provides the basis for superior sensors at low component cost as well as at low system cost including integration and calibration. Combination of different physical (primarily PLD) and chemical (MOFs) nanotechnologies and integration in micro components for efficient mass manufacturing have been demonstrated together with the potential arising from specific electronics and signal processing. The novel approach using nanotechnology for low-cost, partially selective pre-concentration of relevant gases together with nanosensors for their subsequent detection is highly complex and was therefore specifically addressed in the dissemination effort to inform potential end-users as well as the general public. The SENSIndoor video, available on the project website and on YouTube, but also various publications and presentations at international conferences have increased the awareness of this novel solution both in the sensor community, but also in the field of Indoor Air Quality and in potential users in other fields, e.g. medical. With this approach, we have not only provided the technological basis for a new sensor system generation but also addressed the IAQ and other markets and users at an early stage making them aware of the potential created by the technology thus increasing the market pull.

The benefits to the citizens in the European Union are manifold. Three impact levels are specifically reflected here: health, energy savings and creation of a new European and international market.

Impact on health for European citizens
Healthy indoor environments in offices and work spaces, in private homes (especially bedrooms) as well as in public buildings, where people now spend most of their time and are thus most susceptible to

hazardous compounds in the atmosphere, can be achieved based on the novel sensor systems developed within SENSIndoor. According to studies within EU projects like EnVIE 2 Mio healthy life years are lost annually in the EU due to indoor exposure to hazardous compounds. Increased ventilation is therefore demanded with strategies for their implementation having been developed e.g. in the EU project HealthVent. However, the key for implementing these improved strategies was and still is missing today, i.e. sensor systems that can assess the Indoor Air Quality in real time to monitor and control the ventilation and air treatment. Note that a comprehensive ventilation strategy will actually require measurement of air quality not only indoors, but also in the ambient outside, so that the IAQ is actually improved with ventilation. This is due to two issues: first, simply increasing ventilation would greatly increase the energy consumption of the building and, second, many hazardous compounds, especially benzene, are actually introduced to the indoor environment primarily through ventilation. Benzene and other VOCs can be addressed for the first time with the technologies developed within the project at the required low detection limits at ppb or even sub-ppb levels. The availability of VOC specific sensor systems can also lead to new markets in quality control of manufacturing processes, e.g. for building materials, allowing a more effective control of existing standards, and for mitigation strategies beyond simple ventilation such as filtering.

Impact on energy savings in the building segment

Optimized ventilation strategies can greatly reduce power consumption in buildings leading to reduced emission of greenhouse gases as well as lower costs for the citizens. Based on sensor systems able to measure relevant VOCs in real time in different scenarios, threshold limit values for hazardous VOC can be defined effectively for the first time. The energy consumption of houses and buildings taking into account the whole life cycle is responsible for 40% of total EU energy consumption and is the main contributor to greenhouse gas emissions with approx. 36% of the EU's total CO2 emissions according to the EU PPP on Energy efficient Buildings (EeB). Estimates of the impact of demand controlled ventilation (DCV) vary between 25 and 50% of the building energy consumption. Thus, DCV alone can reduce the total energy consumption by 10 to 20%. However, raising this potential requires simultaneous attention to health effects of indoor air. The Sick Building Syndrome is a powerful warning that airtight buildings with ventilation only governed by energy considerations is a step in the wrong direction. Only online monitoring and control of IAQ will allow full exploitation of the potential of DCV achieving acceptance of this technology by the end user. Optimized control of IAQ can achieve a fast return of investment for the sensor systems due to the energy savings.

Note that reduced energy consumption at the level addressed with demand controlled ventilation, i.e. 10 to 20% of the total energy consumption, will have a hugely positive long-term impact on the environment as defined in the EU 20-20-20 goal set by the EC. Buildings are a key component to this goal and especially improvements in existing buildings are required to achieve this goal as outlined by the EU PPP EeB. SENSIndoor has addressed one of the key R&D priorities defined by EeB by contributing to the development of ICTs for energy-smart buildings.

Creation of a new European and international market with huge leverage effect

The market potential directly addressed by SENSIndoor is already huge due to the stock of 160 Mio buildings in the EU according to EeB. Even equipping only a fraction of the rooms in these buildings leads to the demand for millions of smart sensor systems (at a piece cost of around 100 €) every year creating an annual market volume of at least several hundred Mio €. The industrial partners will now enter this market themselves offering new integrated sensor solutions as well as components (sensor elements, pre-

concentrators), processes and services considerably strengthening their individual market position. In addition, to achieve the desired impact in terms of health benefits and reduced energy consumption in this huge market, the technology can also be licensed to large players on the European and international markets. Discussions with potential user are on-going based on the presentations at major conferences, in the SENSIndoor final meeting and direct contacts established via the various SENSIndoor dissemination activities (website, publications, press releases etc.)
Furthermore, the total market created is much bigger due to the leverage effect of these sensor systems: equipment for building monitoring and control, especially for demand controlled ventilation, has a much higher cost, but depends on the information provided by the IAQ sensor nodes. To achieve a noticeable impact within the next years in terms of energy savings and improved health, a concerted effort is required to install optimized DCV in new and existing buildings. SENSIndoor will continue to closely interact with the HVAC industry, primarily through its Advisory Board, to achieve a fast uptake of the new technology. This market is not only addressed in Europe, but worldwide due to similar concerns for health and energy consumption everywhere around the globe. Thus, European manufacturers of both sensor systems as well as building technology achieve a competitive edge in a market worth billions of € per year creating revenue and more jobs in Europe.
In addition, SENSIndoor technology has the potential to address similar markets such as early warning against terrorist threats or industrial accidents, other indoor applications like early fire detection, but also medical applications, e.g. cancer screening based exhaled breath. For these markets with similarly huge potential, further R&D will be initiated based on the SENSIndoor approach additionally strengthening European competitiveness in a worldwide perspective.

European approach
The potential described here could, however, only be achieved with a concerted effort bringing the best expertise in the field together and taking the required mass production at low cost into account within the project. Therefore, SENSIndoor brought together sensor technologies with low-cost potential, i.e. micromachined MOS sensors and integrated SiC GasFETs, and combined these with novel deposition processes suitable for wafer scale processes both for gas sensitive layers as well as for novel pre-concen¬trators. These components have been combined with modular hard- and software components using proven integration technologies by innovative SMEs already active in the target markets. This port¬folio of technologies and expertise could only be found on a European level with partners from five countries.
The European approach was further strengthened by interfacing closely with other EU initiatives and projects. Projects like INDEX, EnVIE and HealthVent as well as the EeB initiative have prepared the rationale for the SENSIndoor approach allowing fast identification of priority application scenarios. On the basis of the existing VOC-IDS project, which addressed the same market but with a more conventional approach the partners have concentrated their efforts on developing the novel nanotechnological components and systems. SENSIndoor has greatly benefited from VOC-IDS with respect to the experience with electronics development as well as building system integration using e.g. KNX or EnOcean interfaces. During the project duration, SENSIndoor closely interacted with current European initiatives and projects, e.g. the COST network EuNetAir, the European Sensor Systems Cluster (ESSC), the sister projects IAQSense and MSP. To this end, the project provided resources for clustering activities in relation to promoting its dissemination and helping in its exploitation activities. Finally, the Advisory Board comprising both health standard representatives as well as industrial partners from the HVAC and

building control industry has allowed SENSIndoor to take current developments in regulations and recommendations continuously into account and address the market at an early stage to ensure a fast take-up of the developed technologies after completion of the project.

Standardization

Interaction with other EU projects and initiatives has also been a key factor for SENSIndoor concerning standardization which was addressed in three aspects:

• Standardization of target VOCs and threshold limit values

Based on EU projects and initiatives, the most relevant target VOCs have been identified and limit values are being set (both on national and on the European levels) or at least recommendations were made for acceptable indoor levels. These regulations and recommendations have formed the basis for the SENSIndoor technology to ensure acceptance by the end users.

• Standardization of interfaces for integration in building control systems

Within the VOC-IDS project the existing standards for both KNX as well as EnOcean interfaces have already been expanded to allow information on total air quality as well as concen¬tration levels of specific VOCs to be passed to the central building control. Decisions on the best ventilation strategy can thus be made locally, i.e. by the individual sensor system, but also in a centralized system. The latter allows application specific ventilation strategies which not only takes the local (room by room) information into account but also global building information.

• IAQ Man machine Interface

Within the collaborative EUproject FEROP, NanoSense defined man machine interfaces in order to assess IAQ quality at working or living places based on consequences and not on concentrations. Consequences are categorized in 4 topics: Health, comfort, quality of sleep and productivity (based on cognitive performance). In order to moderate the setting, related energy consumption is expressed in monetary value. IAQ setting and monetary expression profiles did not exist and had to be standardized by the EnOcean alliance.

• Testing of VOC sensor systems

SENSIndoor was closely linked with the EMRP project KEY-VOCs (Metrology for VOC indicators for air pollution and climate change) via the researcher excellence grant for Dr. Tilman Sauerwald, USAAR-LMT. This allowed an exchange with metrology researchers that are currently defining the required standards for VOC sensor systems. In this context, the SENSIndoor partners have also provided two different test systems for a long term benzene test at JRC Ispra. The results of these tests not only demonstrate the achieved performance to researchers in VOC metrology who are mainly working with analytical methods today, they also greatly support the project´s dissemination activities by providing independent test results.

Steps to bring about the success

The focus of SENSIndoor was clearly on nanotechnology for the sensor layers and pre-concentrator plus integration with microcomponents into complete systems. The project had a very clear focus deriving the scientific and technical achievements from the market and manufacturing requirements. Thanks to the extensive experience of the various partners in their fields, i.e. physical and chemical nano¬technology, microtechnologies for system components, sensor operation and signal interpretation, sensor system integration and calibration, the project could effectively address a highly demanding application with a huge impact in terms of health benefits, reduced energy consumption and economic potential. Both the demand side as well as later market introduction were integrated from the very beginning in the frame of the Advisory Board. Thus, the project partners have received clear input to guide the development throughout the project duration ensuring fast market access after successful completion. This is already evident now from the various discussions and spin-off projects with potential end-users which have been started immediately after completion of the project to continue along the path to market introduction of the novel sensor systems.

Main dissemination activities and exploitation of results

During the SENSIndoor project, dissemination activities played a key role in order to raise widespread awareness of the research conducted in the project as well as initiate and foster cooperation and exchange with research communities, interested commercial parties, such as HVAC systems integrators inside and outside of the EU. Apart from researchers and industrial stakeholders, the dissemination efforts in SENSIndoor targeted the wider general public, aiming at raising awareness for indoor air quality and for the energy savings achievable without risking adverse health effects. Main dissemination activities were:

Website:
The public website of the project (http://www.sensindoor.lmt.uni-saarland.de/) was used as the predominant communication channel to disseminate the project concept, objectives, background information, publications, project achievements and further non-confidential information.

Dissemination Materials:
A foldable flyer in a handy format was designed at the start of the project and packs of it were handed out to the SENSIndoor partners at the kick-off meeting to be distributed at conferences, workshops, trade fairs and other events as well as at the partner institutions.
To achieve higher visibility at meetings, fairs, workshops and/or other events where SENSIndoor was promoted, a so-called eye-catcher poster was designed.
The SENSIndoor partners produced two newsletters in the course of the project, one near the end of year two and the other towards the end of the project. The newsletters contained up to date information on the project progress, on project outputs and general application areas of the sensor system to be developed in SENSIndoor. The newsletters were available for download on the project website, but were also directly distributed via special mailing lists and via the partner institution websites. Hard-copies were alos distributed at several occasions such as the SENSOR+TEST trade fair, Indoor Air conference 2016 and Eurosensors 2016 as well as via the partner institutions.

Project Clip:
During the final year of the project, SENSIndoor produced a project clip which aims at raising awareness for Indoor Air Quality in general but also and especially for the targeted application of Demand Controlled Ventilation. It informs about major progress concerning the development of novel nanotechnology based intelligent sensor systems to be used to measure the quality of indoor air. The clip was distributed via the project website, the EC CORDIS website, via partner websites as well as public video channels such as Youtube.

Publications:
SENSIndoor partners successfully published project-related content in high-impact peer-reviewed journals

and conference proceedings. Wherever possible, joint publications were made. The consortium strived for as many open access publications as possible to maximize scientific outreach. A total of 29 articles in scientific journals and conference proceedings have been published by SENSindoor partners so far:

Peer-reviewed journal publications:

• Andreas Schütze, Tobias Baur, Martin Leidinger, Wolfhard Reimringer, Ralf Jung, Thorsten Conrad, Tilman Sauerwald: Highly Sensitive and Selective VOC Sensor Systems Based on Semiconductor Gas Sensors: How to?
Environments Vol 4, Issue 1, tbd, doi: 10.3390/environments4010020

• Manuel Bastuck, Donatella Puglisi, Joni Huotari, Tilman Sauerwald, Jyrki Lappalainen, Anita Lloyd Spetz, Mike Andersson, Andreas Schütze: Exploring the selectivity of WO3 with Iridium catalyst in an ethanol/naphthalene mixture using multivariate statistics
Thin Solid Films Special Issue SGS 2015, doi:10.1016/j.tsf.2016.08.002

• Caroline Schultealbert, Tobias Baur, Andreas Schütze, Stefan Böttcher, Tilman Sauerwald:
A novel approach towards calibrated measurement of trace gases using metal oxide semiconductor sensors Sensors and Actuators B - Chemical 239, 390–396, doi:10.1016/j.snb.2016.08.002

• Donatella Puglisi, Jens Eriksson, Mike Andersson, Joni Huotari, Manuel Bastuck, Christian Bur, Jyrki Lappalainen, Andreas Schuetze, and Anita Lloyd Spetz: Exploring the Gas Sensing Performance of Catalytic Metal/Metal Oxide 4H-SiC Field Effect Transistors
Materials Science Forum 858 (2016), 997-1000, doi:10.4028/www.scientific.net/MSF.858.997

• Martin Leidinger, Max Rieger, Tilman Sauerwald, Christine Alépée, Andreas Schütze: Integrated pre-concentrator gas sensor microsystem for ppb level benzene detection
Sensors & Actuators B: Chemical 236, 988-996, doi:10.1016/j.snb.2016.04.064

• Joni Huotari, Ville Kekkonen, Tomi Haapalainen, Martin Leidinger, Tilman Sauerwald, Jarkko Puustinen, Jari Liimatainen, Jyrki Lappalainen : Pulsed Laser Deposition of Metal Oxide Nanostructures for Highly Sensitive Gas Sensor Applications
Sensors and Actuators B: Chemical 236, 978-987, doi: 10.1016/j.snb.2016.04.060

• Martin Leidinger, Joni Huotari, Tilman Sauerwald, Jyrki Lappalainen, Andreas Schütze: Selective Detection of Naphthalene with Nanostructured WO3 Gas Sensors prepared by Pulsed Laser Deposition
Journal of Sensors and Sensor Systems 5, 147-156, doi:10.5194/jsss-5-147-2016

• Ville Kekkonen, Saumyadip Chaudhuri, Fergus Clarke, Juho Kaisto, Jari Liimatainen, Santhosh Kumar Pandian, Jarkko Piirto, Mikael Siltanen, Aleksey Zolotukhin: Picosecond pulsed laser deposition of metal-oxide sensing layers with controllable porosity for gas sensor applications
Applied Physics A Vol. 122/Issue 3, 232-238, doi:10.1007/s00339-016-9760-0

• Christian Bur, Manuel Bastuck; Donatella Puglisi; Andreas Schütze; Anita Lloyd Spetz; Mike Andersson: Discrimination and Quantification of Volatile Organic Compounds in the ppb-Range with Gas Sensitive SiC-FETs Using Multivariate Statistics
Sensors and Actuators B - Chemical 214, 225–233, doi:10.1016/j.snb.2015.03.016

• Donatella Puglisi, Jens Eriksson, Christian Bur, Andreas Schuetze, Anita Lloyd Spetz, and Mike Andersson: Catalytic metal-gate field effect transistors based on SiC for indoor air quality control
Journal of Sensors and Sensor Systems 4, 2015, 1-8, doi:10.5194/jsss-4-1-2015

• Donatella Puglisi, Jens Eriksson, Christian Bur, Andreas Schütze, Anita Lloyd Spetz, and Mike Andersson: Silicon carbide field effect transistors for detection of ultra-low concentrations of hazardous volatile organic compounds

Mat. Sci. Forum 778-780, 1067-1070, doi:10.4028/www.scientific.net/MSF.778-780.1067
PhD thesis:
• Christian Bur: Selectivity Enhancement of Gas Sensitive Field Effect Transistors by Dynamic Operation, Linköping Studies in Science and Technology. Dissertations 1644, doi:10.3384/diss.diva-114670 (Co-tutelle thesis between Saarland University and Linköping University, C. Bur received various awards for this thesis: best thesis in measurement science in Germany in 2015, best thesis of the faculty natural sciences and engineering at Saarland University in 2016)
Conference presentations:
Special session Eurosensors 2016
• Martin Leidinger, Tilman Sauerwald, Christine Alépée, Andreas Schütze: Miniaturized integrated gas sensor systems combining metal oxide gas sensors and pre-concentrators
Eurosensors / Procedia Engineering Vol. 168, 293-296, doi: 10.1016/j.proeng.2016.11.199
• M. Bastuck, W. Reimringer, T. Conrad, A. Schütze: Dynamic multi-sensor operation and read-out for highly selective gas sensor systems
Eurosensors / Procedia Engineering Vol. 168, 1685-1688, doi: 10.1016/j.proeng.2016.11.490
• Joni Huotari, Ville Kekkonen, Jarkko Puustinen, Jari Liimatainen, Jyrki Lappalainen: Pulsed laser deposition for improved metal-oxide gas sensing layers
Eurosensors / Procedia Engineering Vol. 168, 1066-1069, doi: 10.1016/j.proeng.2016.11.341
• Isabel Wilhelm, Max Rieger, Jürgen Hürttlen, Michael Wittek, Martin Leidinger, Tilman Sauerwald, Christine Alépée: Novel low-cost selective pre-concentrators based on metal organic frameworks
Procedia Engineering 168, 151-154, doi: 10.1016/j.proeng.2016.11.186
Indoor Air 2016 (joint special session with project IAQSense):
• Eberhard Seitz, Claude Iroulart, Andreas Schütze: Changing the game in the management of Indoor Air Quality - Real time monitoring for improved health, comfort and energy efficiency
Proceedings Indoor Air 2016, 14th international conference of Indoor Air Quality and Climate, July 3-8, summary of session 25, ISBN-13: 978-0-9846855-5-4
• Donatella Puglisi, Manuel Bastuck, Mike Andersson, Joni Huotari, Jyrki Lappalainen, Andreas Schütze, Anita Lloyd Spetz: Gas sensitive SiC-FET sensors for indoor air quality control
Proceedings Indoor Air 2016, 14th international conference of Indoor Air Quality and Climate, July 3-8, paper 780, ISBN-13: 978-0-9846855-5-4

• Martin Leidinger, Tilman Sauerwald, Thorsten Conrad, Andreas Schuetze, Wolfhard Reimringer, Christine Alepee, Max Rieger: Gas measurement system for indoor air quality monitoring using an integrated pre-concentrator gas sensor system
Proceedings of 6. GMM-Workshop, tbd, ISBN: 978-3-8007-4278-3
• Donatella Puglisi, Jens Eriksson, Manuel Bastuck, Mike Andersson, Andreas Schuetze, Anita Lloyd Spetz: Gas Sensors for Indoor Air Quality
AMA-Science Proceedings, 6th Scientific Meeting EuNetAir, Prague, Czech Republic, 05-07 October 2016, 22-25, doi: 10.5162/6EuNetAir2016/06
• Martin Leidinger, Tilman Sauerwald, Christine Alépée, Max Rieger, Andreas Schütze: Integrated pre-concentrator gas sensor system for improved trace gas sensing performance
IEEE SENSORS 2016 30.10.2016-02.11.2016 tbd, doi: 10.1109/ICSENS.2016.7808668
• Martin Leidinger, Max Rieger, Tilman Sauerwald, Christine Alépée, Andreas Schütze: Integriertes Präkonzentrator-Gassensor-Mikrosystem zur Detektion von Spurengasen in Innenraumluft

18. GMA/ITG-Fachtagung Sensoren und Messsysteme 2016, May 10-11, 274-280, doi:10.5162/sensoren2016/4.2.4

• Martin Leidinger, Tilman Sauerwald, Andreas Schütze, Max Rieger, Christine Alépée: Integrated pre-concentrator gas sensor micro system for trace gas detection
12. Dresdner Sensor-Symposium 2015 Proceedings Chapter 5, 72-77, doi: 10.5162/12dss2015/5.4

• Joni Huotari, Jyrki Lappalainen, Jarkko Puustinen, Tobias Baur, Christine Alépée, Timo Haapalainen, Samuli Komulainen, Juho Pylvänäinen, Anita Lloyd Spetz: Pulsed laser deposition of metal oxide nanoparticles, agglomerates, and nanotrees for chemical sensors
Procedia Engineering Volume 120, 2015, 1158–1161, doi:10.1016/j.proeng.2015.08.745

• Mike Andersson, Bo Hammarlund, Anita Lloyd Spetz, Donatella Puglisi: Silicon Carbide Sensor Systems for Harsh Environment Market Applications
AMA-Science Proceedings Fourth Scientific Meeting EuNetAir, 3-5 June 2015, 32-35, doi: 10.5162/4EuNetAir2015/09

• Andreas Schütze, Martin Leidinger, Tilman Sauerwald, Max Rieger, Christine Alépée, Bastian Schmitt: A novel low-cost pre-concentrator concept to boost sensitivity and selectivity of gas sensor systems
IEEE Sensors conference 01.-04.11.2015 1-4, doi:10.1109/ICSENS.2015.7370361

• Martin Leidinger, Max Rieger, Tilman Sauerwald, Marco Nägele, Jürgen Hürttlen, Andreas Schütze: Trace gas VOC detection using metal-organic frameworks micro pre-concentrators and semiconductor gas sensors
Procedia Engineering 120, 1042–1045, doi:10.1016/j.proeng.2015.08.719

• Martin Leidinger, Joni Huotari, Tilman Sauerwald, Jyrki Lappalainen, Andreas Schütze: Nanostructured WO3 Semiconductor Gas Sensor for Selective Detection of Naphthalene
AMA SENSOR 2015 conference May 19-21 2015, 723 - 728, doi:10.5162/sensor2015/E8.2

• Manuel Bastuck, Christian Bur, Tilman Sauerwald, Anita Lloyd Spetz, Mike Andersson, Andreas Schütze: Quantification of Volatile Organic Compounds in the ppb-range using Partial Least Squares Regression
Proceedings SENSOR 2015 May 19-21 2015, 584 - 589, doi:10.5162/sensor2015/D5.1

• Manuel Bastuck, Martin Leidinger, Tilman Sauerwald, Andreas Schütze: Improved Quantification of Naphthalene using non-linear Partial Least Squares Regression
ISOEN 2015, Dijon, France, June 29 - July 1 2015, tbd

Conferences, trade fairs and workshops:
In addition to scientific journals, the SENSIndoor partners disseminated the project outputs via active participation in international scientific conferences, workshops as well as trade fairs, where commercial stakeholders can be addressed directly. Highlights of conferences, workshops and trade fairs with contributions from SENSIndoor partners include: IMCS, Eurosensors, IEEE Sensors Conference, SENSOR+TEST trade fair and accompanying sensor conference, Indoor Air Conference, ICSCRM, ECSCRM, ACHEMA trade fair, FunMAT Annual Conference, Transducers, AFM Conference, SenseAir, EMRS Spring Meetings, MRS Fall Meetings, FOX Workshop, NACSiC Workshop, Sensors and others. The consortium succeeded in organising a special session at EUROSENSORS XXX conference held in Budapest from 04 to 07 September 2016, a highly relevant conference in the field. The session was titled "Nanotechnology-based multi-sensor systems for Indoor Air Quality – Real time monitoring for improved health, comfort and energy efficiency" and included 7 presentations from SENSIndoor partners. In addition, a joint special session was organized at Indoor Air 2016 (a world leading conference on IAQ with

+1,000 participants) in collaboration with project IAQSense titled “Changing the game in the management of Indoor Air Quality – Real time monitoring for improved health, comfort and energy efficiency”. Two partners plus a member of the advisory board and the project PTA contributed to this session; in addition, a summary was prepared for the conference proceedings.
Partners used this opportunity to introduce the functionality and technology of the IAQ sensors developed within SENSIndoor, thus disseminating the obtained results to main stakeholders active in the field of IAQ and promoting uptake of results.
In addition, the Final SENSIndoor Meeting, held in Saarbruecken on 14-16 November 2016, included a Public Session open to external participants from industry, academia, and general public.

Clustering activities:
The SENSIndoor consortium actively participated in the EC’s activities targeted at establishing a cross-cutting Engineering and Upscaling Cluster as well as another cluster on Nano-scale Characterization, addressing metrology, instrumentation and sensors as well as standardization activities. A more specific Sensor cluster (ESSC: European Sensor Systems Cluster) has been founded during the project period, which constituted the main area of involvement of the SENSIndoor project. These clustering efforts bring together members of the scientific community as well as industrial/commercial stakeholders, policy makers and representatives of standardization bodies as the main target groups addressed via this form of dissemination.
Specifically, the cooperation with IAQSense and MSP resulted in a joint booth and a joint session at the Sensor+Test fair 2016 and a joint presentation with IAQSense at Indoor Air 2016. The European Sensor Systems Cluster (ESSC) with the working groups focused on Indoor Air Quality (chaired by Prof. Andreas Schütze, USAAR-LMT; COO) and on integration and commercialization (chaired by Olivier Martimort, NanoSense) provided an important channel to raise awareness for IAQ in the general public and interested stakeholders and was used by partners to further broaden their network.

Exploitation of results
The key exploitable results based on the partner’s foreground have been identified in the scope of the Exploitation Strategy Seminar based on questionnaires provided to each partner. The main key exploitable results that have been identified by the SENSIndoor partners include direct commercial exploitation of R&D results and general advancement of knowledge. In the lists below, only non-confidential exploitation pathways are indicated while product developments for specific applications are excluded to ensure confidentiality.
Commercial exploitation of R&D results:
- New low-power and long term stable micro-hotplates for MOX sensors and novel µ-pre-concentrators
- ps Pulsed Laser Deposition process and system
- Optimized sensor read-out circuit (high dynamic range)
- Optimized temperature control circuits for MOX and SiC-FET sensors
- Improved lab calibration for gas sensors and gas sensor systems for VOCs in the ppb range
- Sensor system for VOCs based on SiC-FETs

General advancement of knowledge:
- Coating technologies according to wafer processing for MIP/MOF layers
- Selective materials/layers for selected VOCs
- Metal Oxide (MOX) material development and tailoring of gas sensing properties using Pulsed Laser

Deposition.
• Development of PLD processing and masking technology for exploited platforms.
• Method for realization and operation of a system based on the combination of passive pre-concentrators and gas sensors for sensitive and selective detection of gases in the ppb and sub ppb range
• Development of methods for optimization and characterization of a system based on SiC-FET sensor technology for sensitive and selective detection of VOCs at low ppb or sub-ppb levels
• Further development of in-field calibration
• Optimization of gate material and tailoring of gas sensing properties using Pulsed Laser Deposition

List of Websites:
http://www.sensindoor.lmt.uni-saarland.de/

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Última actualización: 11 Mayo 2017

Permalink: https://cordis.europa.eu/project/id/604311/reporting/es

European Union, 2025