SENSIndoor

NEWSLETTER ISSUE 2

Contents

The SENSIndoor project has received funding from the European Union's Seventh Framework Programme for research, technological development and demonstration under grant agreement No 604311.

Welcome Address

By Andreas Schütze, Project Coordinator

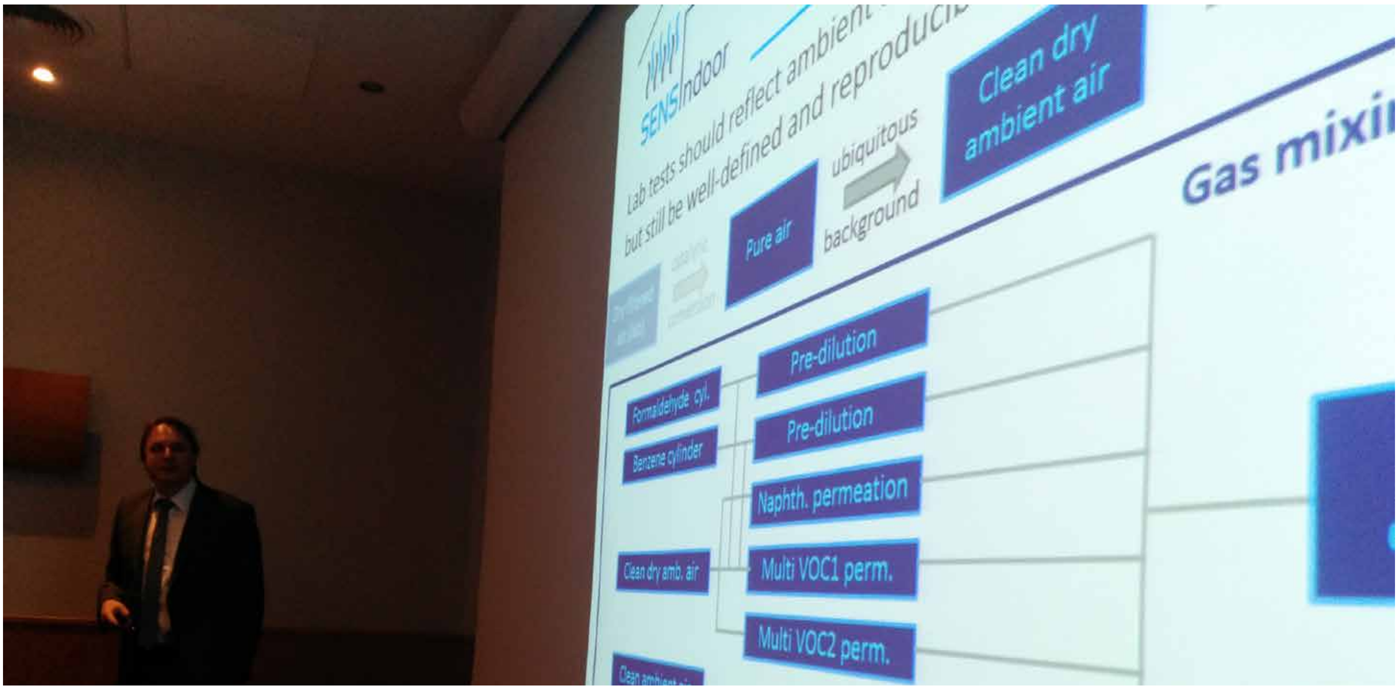
Time flies – it seems like only yesterday that we started together on the path to developing novel, highly sensitive and uniquely selective VOC sensor systems for monitoring of Indoor Air Quality.

Now, we are near the final meeting and have been thinking about the next steps after SENSIndoor for quite some time.

But first, it is time to reflect on excellent project results and on a great project team. Together, we have actually succeeded in bringing low-cost VOC sensors much closer to the market – primarily for IAQ monitoring, but with potential also for industrial and medical applications. And, we have also grown together as a team. Of course, there were some conflicts and problems, but the partners have always succeeded in overcoming the problems and solving their conflicts so that the project was not only successful, but also immensely enjoyable. The project meetings were not seen as a duty, but as a chance to deepen the friendships that have developed over the past years.

Let me briefly touch on the highlights of the project – more details can be found in the specific articles. Oulu University and Picodeon developed PLD-based gas-sensitive layers for both MOS and SiC-FET gas sensors with remarkable sensitivity and selectivity, achieving detection limits down to ppb and sub-ppb levels for our target gases. Linköping University and SenSiC developed novel SiC-FET sensors for VOC detection, and in cooperation with Oulu university the LTCC packaging technology necessary for their application. SGX developed optimized micro-hotplates, not only for the PLD-based MOS sensors, but also for the novel pre-concentrator concept developed in the project. Fraunhofer ICT developed the MOF layers that allow boosting the sensitivity for the target VOCs further to allow successful detection of extremely low target VOC concentrations, not only under controlled lab conditions, but also in the real ambient. Saarland University not only developed novel test methods and systems, but was instrumental in developing the dynamic operating modes for both sensor technologies and for the pre-concentrator, too. NanoSense and 3S developed electronics for the sensor system operation and read-out combining high performance and low cost and studied the system integration and calibration. In fact, the partners

Partners at the 5th SENSIndoor progress meeting.



SENSIndoor presentation at the EUROSENSORS 2016 in Budapest

addressed not only aspects as described in the original project plan, but went beyond this, e.g. with systems supplied for an independent test of benzene detectors carried out at JRC Ispra, or with a novel concept for on-site calibration of VOC sensors at ppb levels.

With the successful research and development came additional goals, mainly disseminating the results further than originally planned. This is reflected not only in the wealth of joint publications in scientific journals and at international conferences, but also in extra efforts during the last project year. While the special session summarising the results at Eurosensors XXX, Europe's leading sensor conference, was planned beforehand, the special session organised at Indoor Air 2016, the world leading conference on IAQ, as well as the exhibition and special session at SENSOR+TEST in Nuremberg were additional activities. These were initiated together with other European projects, MSP and IAQSense and supported by members of our advisory board. Jointly we achieved more attention for the target application – low-cost sensor systems for IAQ monitoring – with much better visibility and impact than from one project alone. Similarly, the activities within the COST network EuNetAir and the cluster ESSC have both stimulated us and benefited from our project. In this context many thanks go to Eberhard Seitz, our PTA. He has been a very constructive partner and provided much positive feedback to our project.

I sincerely hope that we can go the final steps towards commercial success together with the project partners – and perhaps with some of the readers of this 2nd project newsletter. Enjoy reading about the diverse aspects of the SENSIndoor project and contact me or the project partners if we can help you solve your application requirements, if you have ideas to exploit the project results or to develop them further.

Andreas Schütze is full professor in the Department Systems Engineering at Saarland University and head of the lab for measurement technology. He has more than 30 years´ experience in sensor R&D dating back to his PhD thesis on micro gas sensor systems. His research interests include microsensors and microsystems, especially intelligent chemical sensor systems for safety and security, odor assessment and optimized energy systems. He is co-ordinating the SENSIndoor project.

A Word from the Project Officer

By Hans Hartmann Pedersen, Project Officer

SENSIndoor will finish this year as a European project funded by the "Industrial Technologies Programme" in the area of "Advanced Materials and Nanotechnologies".

It has been a pleasure for me to follow the progress of the project, not only for its technical achievements but also due to the excellent and dedicated teamwork shown by the consortium and its professional management.

SENSIndoor has achieved its objectives in developing novel nanotechnology based intelligent sensor systems for selective monitoring of Volatile Organic Compounds as planned and has laid the basis for another EU research and innovation success story.

Downscaling in price and size, the SENSIndoor air quality sensor system makes it possible to develop demand controlled ventilation systems, that can have a high impact in all kind of buildings where people live and work, both on energy consumption and improved air quality.

The SENSIndoor team has widely promoted the project results within both the scientific and industrial communities, and has been actively collaborating with the COST EuNetAir network and the European Sensor System Cluster initiative.

The contributions to the ESSC roadmap are very appreciated for the drafting of future EU research programmes.

The work done to address regulatory aspects is also important. The opportunities for a better control of the indoor environment by using novel intelligent sensor systems, such as the SENSIndoor systems, are important to consider when drafting future regulations that can help combat the sick building syndrome and make our built environment more sustainable.

Great perspectives have been shown by SENSIndoor for the future use of indoor air quality sensor systems.

The EU project will finish soon, but what's more important is that the project team has shown dedication and ability to continue to pursue these perspectives.

I sincerely hope that the SENSIndoor technology will find its rapid way to the market for the best of our citizens and wish the team success with the continued work.

Hans Hartmann Pedersen has been an employee of the European Commission since 1992. Currently he is working in in the department "Research & Innovation" in the Directorate "Key Enabling Technologies", Unit D3 "Advanced Materials and Nanotechnologies". He holds a mechanical engineering degree from the University of Aalborg, Denmark and is SENSIndoor's Project Officer within the EC.

SENSIndoor Technology Highlights

Pulsed Laser Deposition of Advanced Nanostructures for Gas Sensing Applications

By Jyrki Lappalainen, Joni Huotari (University of Oulu, Finland) and Ville Kekkonen (Picodeon, Finland)

Pulsed laser deposition (PLD) is considered to be a relatively novel method to manufacture metal oxide based gas sensing layers. In the deposition method, a high energy laser pulse is shot inside a vacuum chamber at usually rotating target. Energy of the photons of the laser beam is absorbed into the surface layer of the target material forming plasma on the target surface through either laser ablation process, or by evaporation. The plasma, shown in Fig. 1 a), starts to expand adiabatically into the vacuum chamber and, then, condenses on a substrate placed opposite to the target forming a coating layer. A schematic figure of a typical PLD process is presented in Fig. 1 b). A great advantage of PLD method is the straightforward control of film structure by adjustment of the deposition parameters, for example O_2 partial pressure during the deposition and temperature of the substrate. Because the versatility of PLD, it is possible to manufacture different types of materials ranging from epitaxial, high quality thin films, to extremely porous nanoparticle layers. Conventionally, PLD is rather applied with lasers of pulse lengths in the nanosecond range, i.e. ns-PLD, but, nowadays, also more research is done on using picosecond pulsed lasers in PLD, i.e. ps-PLD.

During the SENSIndoor project, both ns-PLD at the University of Oulu and ps-PLD at Picodeon Ltd., have been used to prepare different types of gas sensitive layers on MEMS microheaters provided by SGX Sensortech SA, and, on field-effect transistor (FET) platforms provided by SenSiC AB. A new type of metal oxide layers, formed of small nanoparticles with a tree-like growth mode, has been processed to the sensor platforms [1,2]. The nanotree layers have been shown to be extremely sensitive to different gases, for example tungsten trioxide (WO_3) nanotrees to naphthalene, at least down to 2.5 ppb, with a very good selectivity against benzene and formaldehyde, as well [1,2]. Also, tin dioxde (SnO_2) structures with PLD deposited Pt additives have shown ppb-level of detection of CO and formaldehyde, for example. In Fig. 2 a), a WO_3 nanotree layer deposited by ns-PLD device of University of Oulu is shown, in b) a SnO_2 nanotree layer deposited by ns-PLD is presented, and in c) a WO_3 nanotree layer deposited by ps-PLD device of Picodeon Ltd can be seen [2]. It can be noted that the particle size of the porous layers is extremely small. On the other hand, also denser and thinner WO_3 films have been deposited to the gate area of the FET gas sensor platforms of SenSiC AB to serve as an additional oxide under a sputtered, thin iridium catalytic layer.

It has been shown that the PLD deposited WO_3 thin films increases the selectivity of the FET gas sensor compared to a more conventional sensor with pure Ir gate, and that the FET sensors with the additional WO_3 layer are capable of detecting benzene and naphthalene down to levels of few ppb [3,4].

a)

b)

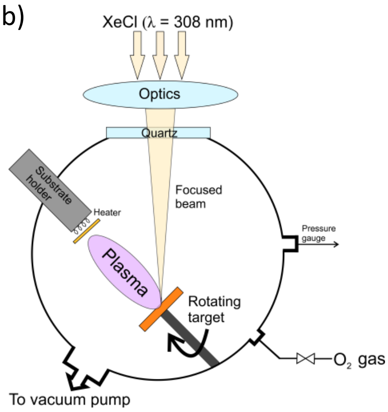


Fig. 1 a) Bluish plasma plume generated in the PLD process, and b) a schematic of a typical PLD system

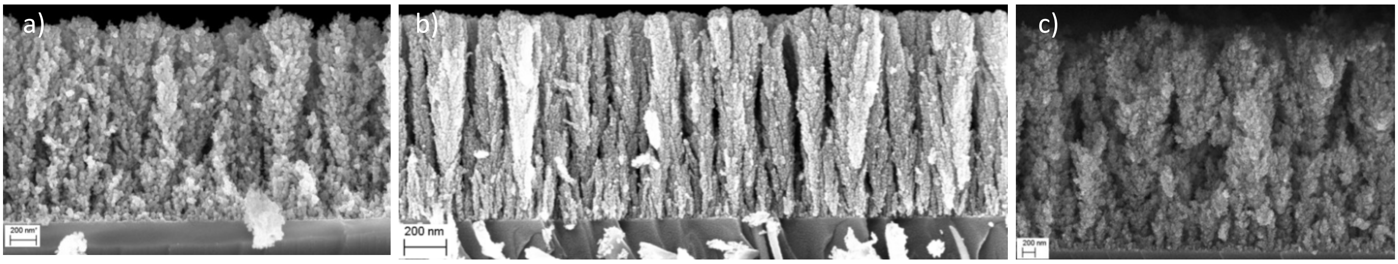


Fig. 2: a) WO_3 nanotree layer deposited by ns-PLD device of University of Oulu. (b) SnO_2 nanotree layer deposited by ns-PLD device of University of Oulu, and c) WO_3 nanotree layer deposited by ps-PLD device of Picodeon Ltd [2].

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Jyrki Lappalainen is Professor of Electronics Manufacturing Technologies in the Microelectronics and Materials Phsysics Laboratories at the University of Oulu, Finland. He is also the head of the Functional Electroceramic Thin Films and Nanostructures research group of University of Oulu(UO-FETF). In SENSIndoor, he is a work package leader and responsible for manufacturing and characterization of nanostructured materials for chemical sensors.

Joni Huotari is working as a PhD student at the University of Oulu, where he graduated in electrical engineering. He is the principal researcher for manufacturing and characterization of nanostructured materials for chemical sensors. Within SENSIndoor, he is the vice work package leader and the vice team leader of the UO-FETF research group in project SENSIndoor.

Ville Kekkonen, M.Sc. (tech), works as a PLD Technologist at Picodeon, being involved in the research and development activities of the company. He has been working with pulsed laser deposition since 2004. Within the SENSIndoor project, he is responsible for the PLD process and the development of sensing layers.

Operating Modes and Signal Processing

By Manuel Bastuck (Saarland University, Germany and Linköping University, Sweden), Donatella Puglisi and Anita Lloyd Spetz (Linköping University, Sweden), Mike Andersson (SenSiC and Linköping University, Sweden)

Besides optimisation of sensing materials and their structure, additional selectivity of the sensor system can be gained with specialised operating modes and signal processing algorithms. Both sensor types, i.e. MOS and GasFET, are driven with temperature cycled operation (TCO), as shown in Fig. 1.

Different temperatures change the sensors' properties and, thus, create an array of virtual sensors which all react differently to certain gases. This approach reduces the sample rate, but increases the dimensionality of each measurement point and, eventually, increases the selectivity drastically. For the MOS sensor, the TCO is complemented with the pre-concentrator (Fig. 1a), selectively adsorbing gas at low temperature and releasing the gas molecules during the high temperature phase. The MOS sensor reaches resistance values in the GΩ range during some parts of its temperature cycle. In order to measure these resistance values reliably and with low noise, a logarithmic amplifier close to the sensor converts the small resulting current into voltages on the order of volts. The GasFET, on the other hand, cannot yet be integrated with the pre-concentrator due to its relatively high heating power. However, its gate bias can be modulated (gate bias cycled operation, GBCO, Fig. 1b), adding another layer of parameter variation and, eventually, increased selectivity as well.

At typical sample rates around 10 Hz, one measurement point, i.e. one cycle, can have 1,000 dimensions and more. This high dimensionality causes mathematical and numerical problems summarised as the "curse of dimensionality". Moreover, many of the virtual sensors will have almost identical properties, providing redundant information against which machine learning algorithms are, in general, not very robust. Hence, the dimensionality is reduced in a first step called feature extraction (Fig. 2) where more than 1,000 features are replaced by only a few parameters describing the cycle shape, e.g. mean and slope. Prior to that it can be beneficial to preprocess the raw data, e.g. to eliminate the influence of humidity for MOS sensors by dividing each cycle by its mean value. The extracted features are further reduced by linear discriminant analysis (LDA), an algorithm which finds a new, lower-dimensional coordinate system where the different classes, i.e. gases, are most compact and most separated from each other. While finding this projection ("training") is computationally relatively costly, projecting new data points ("evaluation") is cheap. Classification of a new data point is easiest done in a one-dimensional system as it can either be below

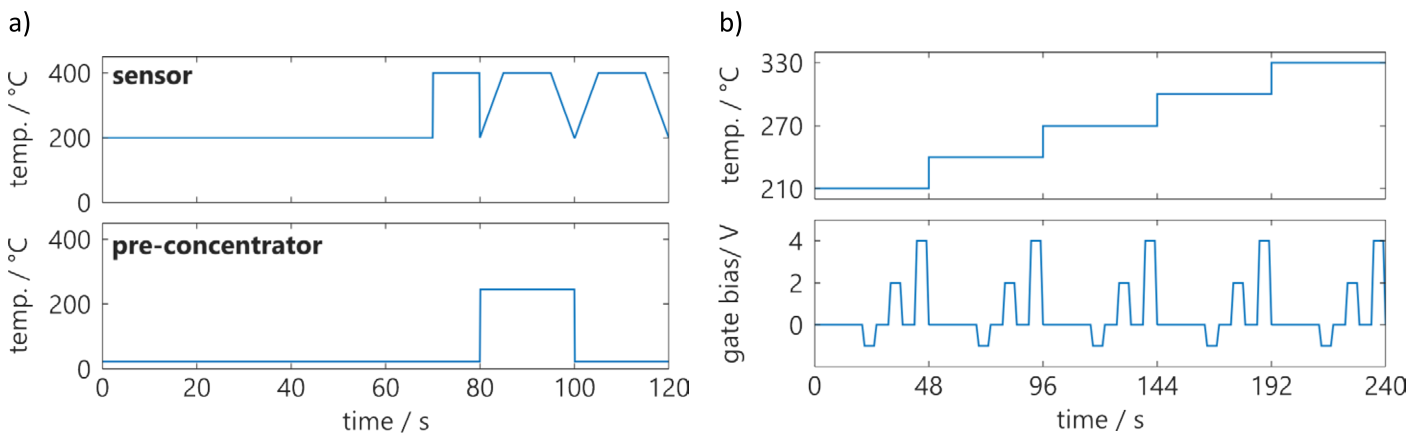

Fig. 1: a) MOS sensor with TCO and temperature modulated pre-concentrator, (b) GasFET with TCO and GBCO.

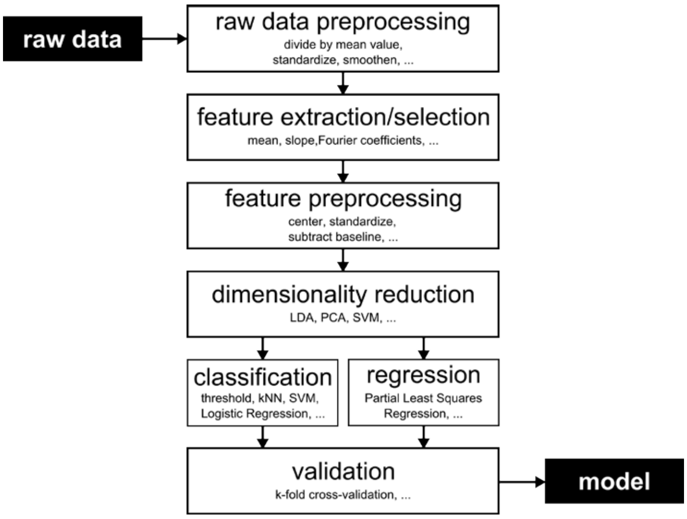

Fig. 2: Model building workflow.

or above a scalar classification threshold. Therefore, we use a series of specialised one-dimensional projections to enable implementation on low-cost hardware. The first projection for example discriminates between low/high humidity. Based on that result, the data point is given to another projection specialised for either low or high humidity, discriminating e.g. ethanol concentration, and so on until a decision is reached whether a certain target gas exceeds the legal limit. Exact quantification is possible by using the same features for a Partial Least Squares Regression (PLSR) model which returns a concentration (instead of a classification) as result. All models are validated using k-fold cross-validation to determine their predictive power and to avoid overfitting to the training data. This technique divides the training data into k parts, builds a model from k-1 parts, predicts the remaining part and compares prediction and actual class. The mean of k repetitions is the average correct classification rate.

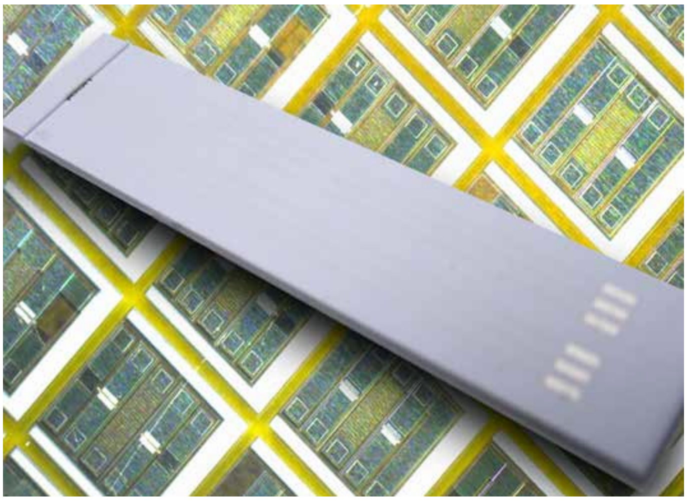

Fig. 3: Smart package solution: GasFET in an LTCC package.

Manuel Bastuck is currently enrolled in a joint PhD program between Saarland University, Germany and Linköping University, Sweden. He graduated in Microtechnology and Nanostructures at Saarland University in 2014. In SENSIndoor, he optimizes the cyclic operation mode for the newest generation of GasFET sensors and develops data fusion and evaluation algorithms for the sensor system.

Donatella Puglisi, PhD in Physics, is Assistant Professor in Applied Sensor Science at Linköping University, LiU, Sweden. Her research interests include sensor processing and characterization of gas sensitive field effect transistors based on silicon carbide for detection of trace amounts of hazardous indoor air pollutants. She is deputy team leader within the SENSIndoor project.

Anita Lloyd Spetz is Professor in Applied Sensor Science at Linköping University, LiU, Sweden and at University of Oulu (UO), Finland. Her research involves SiC-FET high temperature gas sensors for harsh environment, PM (UO) and graphene sensors, smart sensing and data evaluation. She is member of the board of SenSiC AB for commercialization of SiC-FET sensors. She is LiU teamleader in the SENSIndoor project.

Mike Andersson is the CTO of SenSiC AB and associate professor at Linköping University, Sweden. He has a Ph.D degree in Applied Physics and looks back on many years' experience in research and project management. In SENSIndoor, he is work package leader and company representative considering the field effect sensor platform development.

Novel Micro Pre-concentrator

By Christine Alépée (SGX Sensortech, Switzerland), Martin Leidinger and Tilman Sauerwald (Saarland University, Germany), Max Rieger (Fraunhofer Institute for Chemical Technology, Germany), and Wolfhard Reimringer (3S – Sensors, Signal processing, System GmbH, Germany)

In order to reach the ppb-level detection thresholds required for the targeted in-door air quality application, novel micro pre-concentrators (µPCs) were developed to boost the system's sensitivity and selectivity well above the intrinsic ones of stand-alone MOS sensors.

Optimisation of the building blocks

To start with, the elements of these micro pre-concentrators – micro-hotplate and adsorbent – were optimised separately.

A novel dedicated MEMS hotplate was developed. It is characterised by excellent temperature uniformity and heater stability, which are both essential for precise and controlled release of the adsorbed gas. The best trade-off between maximum adsorption/desorption capacity and minimum power consumption had also to be defined. The resulting optimised micro-heater platforms have a footprint of only 5 mm^2 and present an active surface of 0.57 mm^2 that requires only 50 mW to reach a desorption temperature of 200°C. It is important to highlight that a 50 µm thin layer of MOF covering this tiny active surface, thanks to the MOFs' exceptionally high interior surface areas (above 1000 m^2/g), represents an effective adsorption surface of the order of 100 cm^2!

As described in the SENSIndoor Newsletter, Issue 1, the MOF materials chosen as possible sorbent material for the novel micro pre-concentrators – MIL-53, HKUST-1, and UiO66 – were characterised via inverse gas chromatography and mass spectrometry [1] [2]. The adsorption and desorption parameters measured this way for the three tested MOFs served for candidate ranking.

The system's configuration was optimised by FEM simulations. They evidenced that, against all expectations, a complex face-to-face placement of sensor and µPC yielded hardly better pre-concentration factors – but much stronger undesired thermal cross-talk – than the much more straightforward side-by-side configuration, which was therefore eventually retained.

The adsorption and desorption parameters of MOFs were introduced into the side-by-side FEM model of the whole sensor system [2], which showed that the expectable pre-concentration factor of such a system was in the range of 11 to 32, depending on the sorbent material.

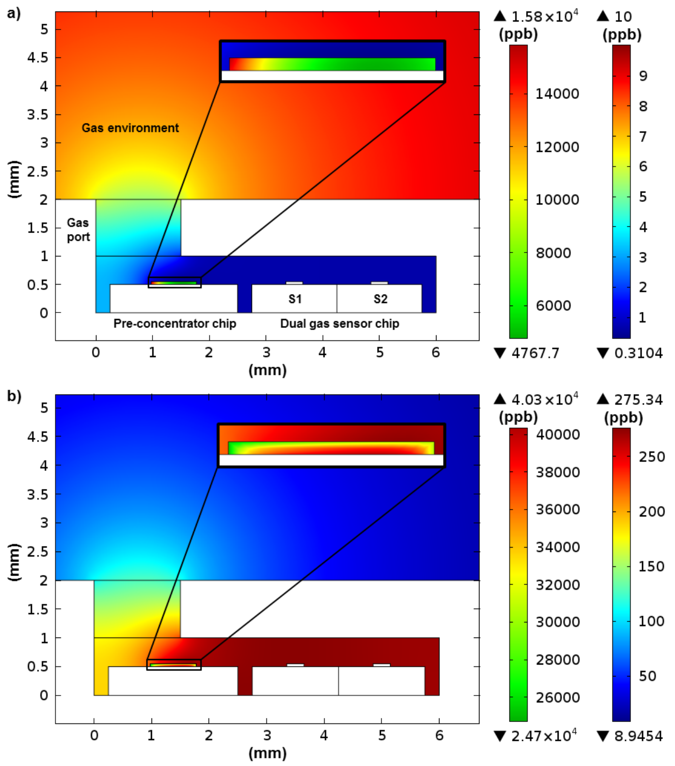

Fig. 1: Simulated gas concentrations inside the pre-concentrator material (left scale) and in the air (right scale), simulated for benzene as target gas and HKUST-1 as pre-concentrator material. (a) 6.5 s after start of adsorption at 50 °C. Nearly all gas from inside the package has been adsorbed in the pre-concentrator material. (b) 1.5 s after start of desorption at 200 °C. The highest gas concentration is obtained inside the microsystem, i.e. at the locations of the two gas sensor chips

Integration

The separately improved elements were then put together to form a miniature µPC+sensor system.

This required first the optimisation of the MOF deposition technique onto the micro-hotplates, with respect to MOF layer diameter and thickness, alignment to the micro-heater structure, adhesion to the micro-heater platform, and, last but not least, scale-up potential and adaptability to the widest possible variety of MOFs. Direct growth, drop dispensing, screen printing, and airbrushing were tested and the latter one became the method of choice as it gave the best technical results and is, in addition, a low cost and high-volume compatible batch process. The optimised airbrushing method currently allows for coating 50 micro-heaters at once with MOF layers up to 50 µm thick and a MOF/heater coverage of about 80% (see Figure 2) and the method is up-scalable to wafer level, i.e. for coating of hundreds of micro-heaters in parallel.

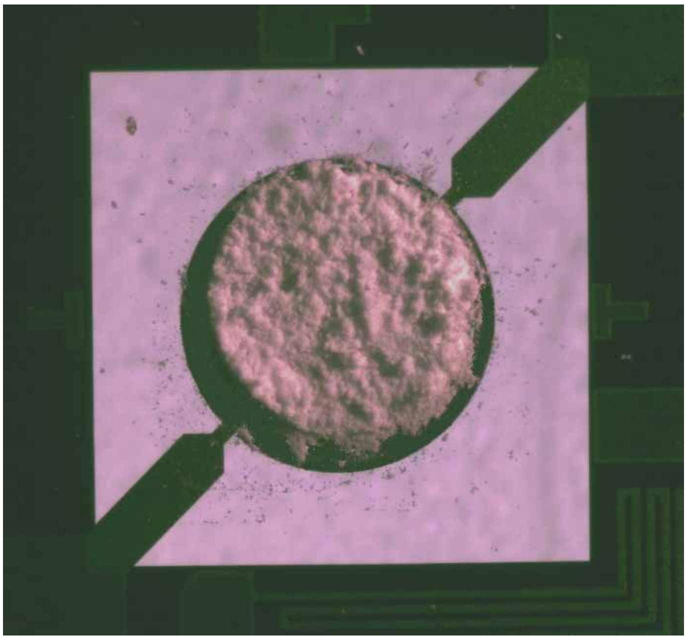

Fig. 2: A MOF-coated micro-heater (heater diameter: 850 µm).

The benzene pre-concentration effect of the MOF materials was verified in test measurements as shown for example for UiO-66 in Figure 3, which clearly shows the significant signal increase obtained thanks to the µPC.

The optimised micro pre-concentrator chips were then integrated according to the configuration recommendations, drawn from the above-mentioned FEM simulations. The µPC chips were mounted next to

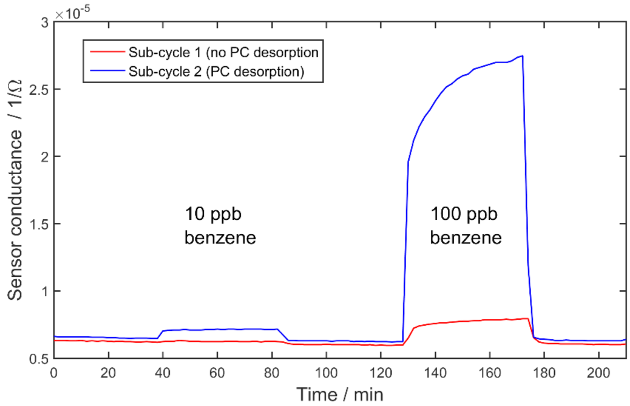

Fig. 3: Signals of a gas sensor mounted next to a µPC. The signals were recorded with the µPC either active or inactive (respectively blue and red curves).

1 or 2 MOS sensor chips inside the cavities of standard 7x5 mm^2 SMD housings, which were eventually capped with a lid presenting a gas inlet above the µPC chip and confining the MOS sensor(s) in the package's dead end (Figure 4). Future ramp-up in volume should be relatively straightforward, as all assembly steps – die bonding, wire bonding, and cap bonding – are standard worldwide as well as in SGX's production line and are high-volume compatible.

Fig. 4: Array of assembled and capped µPC+sensor systems. On the samples without cap, the µPC and a dual MOS sensor are visible (close-up view in Fig. 5).

Testing

The resulting µPC+sensor systems were first evaluated in the lab, mainly for their efficiency for benzene pre-concentration and detection, but also for their cross-sensitivity to other gases and to humidity. The best candidates were then mounted into SENSIndoor control and read-out systems by 3S and NanoSense to be evaluated in the field until November 2016, in the frame of SENSIndoor's final full-size test.

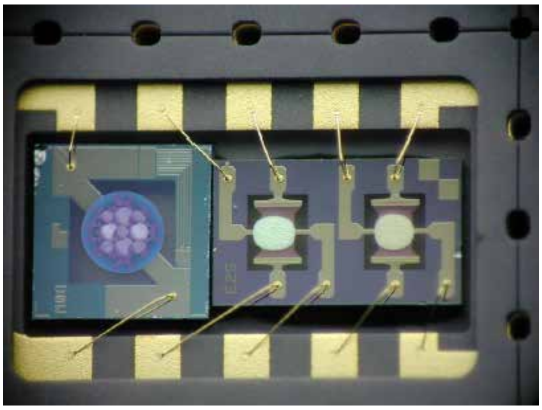

Fig. 5: Top view of the integrated pre-concentrator gas sensor system (without lid). The PC chip is located in the left corner; a commercial SGX dual gas sensor chip is placed next to it.

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Christine Alépée is working as MEMS Development Manager with SGX where she develops and improves commercial gas sensing MEMS. She studied and gained her doctorate degree in micro-engineering at the Swiss federal Institute of Technology in Lausanne. In the frame of SENSIndoor, she is leading the development of the novel MOS sensors and of the pre-concentrator micro-hotplates and acts as link between the technology providers and the system integrators.

Martin Leidinger is pursuing his PhD at the Saarland University, Germany, where he studied mechatronics engineering. His research interests are in design, operation and characterisation of sensor systems for indoor air quality. In SENSIndoor, he evaluates the performance of the PLD MOS gas sensors and sensor systems for trace gas concentrations. He is also involved in the development of the integrated pre-concentrator/ gas sensor microsystems.

Tilman Sauerwald is working as a post-doc at the Lab of Measurement Technology at Saarland University. He received his PhD in Physics, working on the influence of surface reactions to the multi-signal generation of metal oxide sensors. Within the SENSIndoor project, he is workpackage leader for sensor operation modes and data processing as well as for testing and calibration of the systems.

Max Rieger is employed in the department of Energetic Materials of the Fraunhofer Institute for Chemical Technology (ICT), where he is currently finishing his PhD thesis dealing with metal organic frameworks for preconcentration for hazardous materials and explosives. He studied Chemistry at the University of Würzburg, Germany and graduated in 2011. Within the SENSIndoor project, he is concerned with initial MOF characterization and fabrication of layers on top of the sensor systems µ-preconcentrators.

Wolfhard Reimringer is responsible for electronics design and system integration at SENSIndoor partner 3S GmbH. Having worked with this company since its formation, he joined for a full time position after graduating with an M.Sc. in electrical engineering. His thesis covered a field test device for mnt-era.net project VOC-IDS. In SENSIndoor he is working on MOS and SiC-FET field test demonstrators as the designated work package leader of WP8.

Electronics Integration and Log-amplifier Read-out

By Wolfhard Reimringer (3S – Sensors, Signal processing, System GmbH, Germany), Olivier Martimort (NanoSense, France), Tobias Baur and Tilman Sauerwald (Saarland University, Germany)

The SENSIndoor sensor sub-system consists of a combination of one pre-concentrator and two metal oxide semiconductor (MOS) sensors, each on a separate micro hotplate. This sub-system is complemented by dedicated electronics for heater control and sensor read-out, collocated as close as possible to avoid interference and signal loss. The micro hotplates need to be controlled at different defined temperatures; to this end the hotplate heater is used as a PTC resistor for closed loop temperature control either by analogue or digital feedback. Reading out the sensors is a challenging task: It has been shown that fast temperature changes in metal oxide semiconductor sensors lead to increased selectivity, sensitivity and stability in gas detection [1][2]. As the conductance of the sensor is dominated by semiconductor grain boundaries, it shows a strong temperature dependence. Therefore, this operation mode in combination with the gas reaction leads to a variation of the gas sensor's conductance over many orders of magnitude (cf. Fig. 1). A measurement of the sensor conductance at low noise level can be achieved by using amplifiers with logarithmic response.

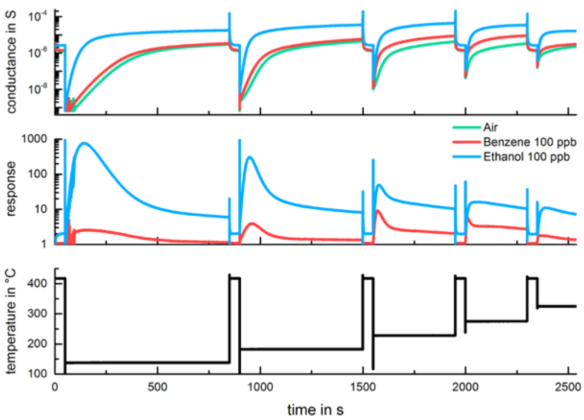

Fig. 1: Gas sensor response in temperature cycled operation (TCO) measured utilising a logarithmic amplifier cf. [1]

As the prospective application should allow to integrate the sensor device into standard building installation, the SENSIndoor field test demonstrator has been designed so compact that it conveniently fits into a flush mount switch box. Fig. 2a shows the electronics by SENSIndoor partner 3S, consisting of a two PCB stack (sensor electronics and controller with power supply) as well as a break-out board with SD card and NDIR-CO_2-Sensor for field test data acquisition.

The system also features an RGB status LED and a sensor for temperature and humidity. It can be powered from 9 to 32 VDC and connects to data interfaces via a general purpose serial interface. Fig. 2b shows the electronics by SENSIndoor partner NanoSense, consisting of a single PCB with UART, I2C and USB interface. The board also includes a temperature and humidity sensor. Size and pinning are similar to NDIR CO_2 modules and both can fit into a 40x40mm flush mount switch box.

Heater control is a combination of analogue temperature

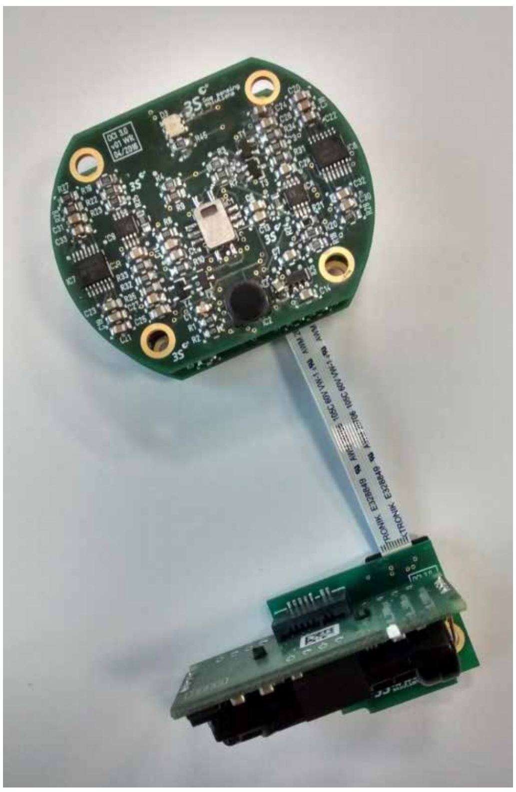

Fig. 2a: Field test electronics demonstrator (3S GmbH), sensor sub system in the center of upper PCB.

Fig. 2b: Field test electronics demonstrator (NanoSense) sensor module aside an NDIR CO_2 module

control – based on the PTC characteristics of the heater material – and digital set point. The analogue control loop provides smooth behaviour which reduces noise coupling into the sensing layer. Digital or analogue feedback allows for temperature control of the heater with a reproducibility of better than 1 °C, independent of ambient temperature or air flow. Sensor read-out is based on an integrated dual logarithmic amplifier (LOG2112, Texas Instruments for 3S and ADL5310 from Analog Device for NanoSense).

The circuit picks up sensor conductance as a current variation which is transformed into a logarithmic voltage response. A reference current allows setting of the impedance range; the ADC resolution coupled with final gain setting becomes then the limiting factor of the graduation of the logarithmic scale. With this set-up a measurement range of e.g. 250 Ω – 1 GΩ with a graduation of several hundred points per decade is achieved.

Wolfhard Reimringer studied electrical engineering and received his M.Sc. in from Saarland University of applied sciences, Saarbruecken, Germany in 2013. He first joined 3S as a developer for electronic hardware in 2009, subsequently taking over additional responsibilities in system integration and design as well as product management tasks for air quality applications. Already having designed the field test device used in the mnt-era.net project VOC-IDS he is designated work package leader for WP8 “Sensor system integration” in SENSIndoor.

Olivier Martimort is a senior engineer in electronics and has been involved in the only French contribution to the US Strategic Defence Initiative (SDI). He set up NanoSense in 2002 and is currently the company’s CEO. He manages the R&D department and is personally involved in each project. He personally designed the electronic of the NanoSense field test sensor module based on a Cortex M4 microprocessor.

Tobias Baur is currently working on his PhD that deals with MOS gas sensor systems for gas chromatography and electrical design of sensor devices. He studied Microtechnology and Nanostructures at Saarland University, Germany and received his M.Sc. at the Lab of Measurement Technology in 2016. In SENSIndoor he is in charge for designing the electronics for high-resistance sensors.

Tilman Sauerwald is working as a post-doc at the Lab of Measurement Technology at Saarland University. He received his PhD in Physics, working on the influence of surface reactions to the multi-signal generation of metal oxide sensors. Within the SENSIndoor project, he is workpackage leader for sensor operation modes and data processing as well as for testing and calibration of the systems.

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Calibration of Sensor Systems

By Caroline Schultealbert, Tilman Sauerwald (Saarland University, Germany), Thorsten Conrad (3S – Sensors, Signal processing, System GmbH, Germany) and Olivier Martimort (NanoSense, France)

The SENSIndoor consortium is aiming at reliable gas sensor systems. Therefore, calibration plays an important role. However, the reproducible generation of gas mixtures in the ppb and sub-ppb range is still very painstaking and not yet available on larger scales. For this reason, techniques for calibration, in-factory as well as ex-factory, have been investigated comprehensively.

In-factory calibration

In factory calibration has been tested in lab using gas mixing devices. Following the line of earlier work, we use high ratio dilution of gases to obtain gases in ppb levels in a reproducible manner without extensive change of background [1]. The upscaling of the gas mixing systems is currently under investigation. For mass production a separate air stream to each sensor cannot be used. The possibility to calibrate between 10,000 and 20,000 sensors at a time (see figure 1), generating VOC via liquid phase injection and vaporization, has been studied. The individual control of each sensor requires a sophisticated bidirectional digital communication system. Since gas sensors are often highly sensitive to other gases and changing ambient conditions, not only the target gases (benzene, naphthalene, formaldehyde) have to be provided to the sensor, but the lab calibration has to cover a good representation of any condition the sensor may be exposed to during its lifetime. This includes humidity variations and several not hazardous VOCs (background gases such as ethanol). The sensor systems dedicated to the field tests have been tested with an overall variation of gas exposures for one week. After that, the measurement data is used to create and validate a model using LDA and PLSR (see page 7, *Operating modes and Signal-Processing*) which will be used during the SENSIndoor field tests. Based on the results of lab and field tests we are aiming at reducing the complexity of the tests to obtain reliable model parameters with shorter test duration.

Fig. 1: NanoSense calibration chamber (2 m³) with a trolley supporting 7 columns of 3 racks on both sides. Each rack has 40 slots which can handle a board with 9 SENSIndoor sensor systems. In total 15,120 systems can be calibrated at a time.

Ex-factory calibration

During an expected lifetime of 10-20 years the system should be checked and recalibrated periodically. At this stage of the SENSIndoor project, the stability of the sensor has only been tested for a short period and, therefore, the required calibration strategies and calibration intervals can only be estimated from typical drift mechanisms for gas sensors. Sensor drift could be caused by heater ageing and sensitive layer ageing. Whereas heater drift can be automatically recalibrated in situ via driving at constant power and known ambient temperature, the calibration of the sensitive layer requires a much more complex procedure that might include test gases. To this end, a transportable calibration standard was developed based on a two-phase thermodynamic equilibrium of a liquid phase with a huge VOC reservoir and a small gaseous phase [2].

The liquid phase consists of a mixture of a non-volatile liquid and a diluted target VOC. GC-MS analyses of the headspace concentration were conducted, proving the traceability of the standards. An isothermal vial utilising a phase change material cladding was developed to keep the temperature of the calibration standard stable for several hours. A tight connection to the sensor is guaranteed by a quick coupling, which is self-closing on the sample's side for security reasons. Using a pure squalane sample, a VOC concentration of zero can be achieved, since all VOCs in the included ambient air are solved without influencing humidity and permanent gases like hydrogen. The calibration was tested under ambient conditions and yielded satisfactory accuracy.

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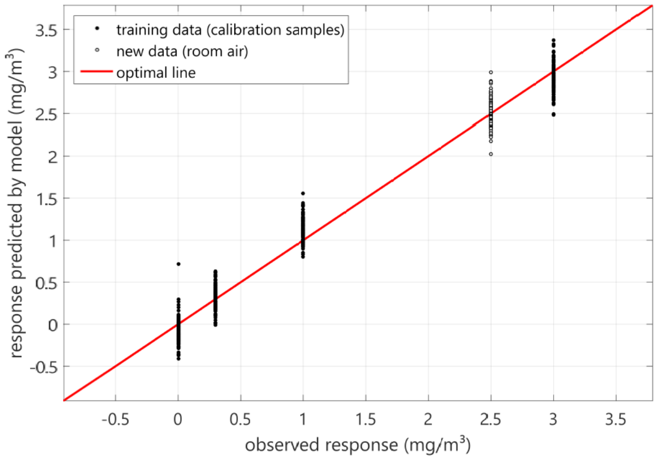


Fig. 2: PLSR model achieved under room conditions using the in-field calibration method and projected office indoor air.

Fig. 3: Calibration standard with (left) and without (right) phase change material cladding.

Caroline Schultealbert is working at the Lab for Measurement Technology of Saarland University, where she is currently working on her PhD, dealing with the improvement of gas sensor performances by temperature cycling and model based feature extraction. She studied Microtechnology and Nanostructures in the Saarland University and graduated in 2015. Within the SENSIndoor project, she is involved in the in-field calibration and testing of the sensors.

Tilman Sauerwald is working as a post-doc at the Lab of Measurement Technology at Saarland University. He received his PhD in Physics, working on the influence of surface reactions to the multi-signal generation of metal oxide sensors. Within the SENSIndoor project, he is workpackage leader for sensor operation modes and data processing as well as for testing and calibration of the systems.

Thorsten Conrad is founder and managing partner of 3S and member of several committees and standardisations groups. After receiving his degree in electrical engineering from Saarland University, Germany, he gained research experience in the Department of Microsystems Engineering (IMTEK) in Freiburg and in the Lab of Measurement Technology in Saarbrücken. In SENSIndoor, his focus is on system integration and calibration strategies.

Olivier Martimort is a senior engineer in electronics and has been involved in the only French contribution to the US Strategic Defence Initiative (SDI). He set up NanoSense in 2002 and is currently the company's CEO. He manages the R&D department and is personally involved in each project. He contributed with his team to the design of a fully digital mass production calibration chamber.

Interfacing Activities

By Corinna Hahn (Eurice, Germany)

The project partners are closely linked to a wider community of established universities and research institutes as well as innovative companies in the field. Collaborations exist at different levels and with different foci.

The EuNetAir COST Action TD1105 entitled "European Network on New Sensing Technologies for Air-Pollution Control and Environmental Sustainability" offered networking opportunities and also represented a forum for SENSIndoor partners to disseminate and discuss their results. The action ends on November 15, 2016 after 4.5 years and is considered a success story. SENSIndoor partners not only actively participated in the meetings, but served as key members – Anita Loyd-Spetz in her function as Co-Chair of the Action and Andreas Schütze as leader of Work package 2.

Collaboration with the European Sensor-Systems Cluster (ESSC)

ESSC (www.cluster-essc.eu) is the European Cluster on Sensor Systems supported and was launched by the EC at Brussels, 27 November 2014. The main objective of the ESSC is to join efforts in order to avoid defragmentation and to promote synergies with industrial leadership and European cooperation in the field of the research and innovation of sensor-systems applications.

Objectives include the identification of common interests in on-going research and development (e.g. open calls, training), the provision of a forum for discussion, problem solving and analytical planning R&D activities in Europe, the establishment of an EU-wide meeting platform for researchers, industry and end-users and the dissemination of sensor-related issues/findings to stimulate awareness for the invisible environmental problems and support citizen science.

SENSIndoor partners involved in ESSC include Saarland University, NanoSense and EURICE.

In October 2016, the cluster partners published a "Roadmap Towards European Leadership in Sensor Systems" which represents a contribution to define PRIORITIES of a Roadmap for the European Commission (primarily DG Research & Innovation Directorate Key Enabling Technologies - Unit Advanced Materials and Nanotechnologies, but also other units) useful to select topics for future H2020 Calls (2018-2020).

Joint exhibit and sessions

SENSIndoor partners have organised a number of events in close collaboration with the ESSC and related projects EuNetAir (www.cost.eunetair.it) , MSC (www.multisensorplatform.eu) and IAQSense (www.iaqsense.eu). Such events include conference sessions at the Indoor Air 2016 (Special session 25 on July 07, 2016) and the EUROSENSORS 2016 (Special session on September 07, 2016) as well as a common booth at the Sensor+Test Fair 2016.

The SENSIndoor consortium presented itself on a joint exhibition with MSP and IAQSense at the Sensor+Test Fair 2016. The joint booth attracted many interested visitors, both, from industry and science. Prototypes of four ultra-sensitive sensors and sensor systems were presented leading to various contacts to potential clients from the HVAC industry and other branches. Thus, the conference offered a perfect chance for networking, as the project is slowly moving towards its end.

Corinna Hahn is Senior Programme Manager at Eurice GmbH. She has over 15 years of experience in EC funded projects, with a particular focus on technical sciences, energy and climate. In SENSIndoor, she is leading the work package on dissemination and exploitation of project outcomes, including interfacing with sister projects and networks, awareness raising and common actions.

Key Publications

Pulsed Laser Deposition (PLD)

Pulsed laser deposition of metal oxide nanostructures for highly sensitive gas sensor applications

J. Huotari, V. Kekkonen, T. Haapalainen, M. Leidinger, T. Sauerwald, J. Puustinen, J. Liimatainen, J. Lappalainen

Sensors and Actuators B: Chemical 236, 978-987, 2016, DOI:10.1016/j.snb.2016.04.060.

Abstract: Nanosecond and picosecond pulsed laser deposition (PLD) was used to prepare metal oxide nanostructures with different morphologies as gas sensing materials on top of oxidized silicon substrates and commercial SGX Sensortech SA MEMS microheater platforms. The layers were formed of different types of nanostructures including nanoparticles, agglomerates, and nanotrees with fractal-like growth. Clear dependencies between the deposition parameters, structural morphology, and gas sensing performance were found. Also, some differences in the morphologies of the layers were seen when picosecond PLD was used instead of nanosecond PLD. Many of the sensing materials were found to be highly sensitive to different types of gaseous species. We investigated inorganic gases in the ppm range (2–400 ppm) including NO, CO, and NH3, and the selectivity and sensitivity were shown to be dependent, not only on layer morphology, but also on the measurement temperature. Moreover, an investigation with volatile organic compound gases in the ppb range demonstrated that WO_3 layers are highly sensitive and selective towards naphthalene at least down to 2.5 ppb.

MOS microsensors for ppb level VOC

Selective detection of naphthalene with nanostructured WO_3 gas sensors prepared by pulsed laser deposition

M. Leidinger, J. Huotari, T. Sauerwald, J. Lappalainen, A. Schütze

Journal of Sensors and Sensor 5, 147-156, 2016, DOI:10.5194/jsss-5-147-2016.

Abstract: Pulsed laser deposition (PLD) at room temperature with a nanosecond laser was used to prepare WO_3 layers on both MEMS microheater platforms and Si/SiO_2 substrates. Structural characterization showed that the layers are formed of nanoparticles and nanoparticle agglomerates. Two types of layers were prepared, one at an oxygen partial pressure of 0.08 mbar and one at 0.2 mbar. The layer structure and the related gas sensing properties were shown to be highly dependent on this deposition parameter. At an oxygen pressure of 0.2 mbar, formation of ε-phase WO_3 was found, which is possibly contributing to the observed increase in sensitivity of the sensor material. The gas sensing performance of the two sensor layers prepared via PLD was tested for detection of volatile organic compounds (benzene, formaldehyde and naphthalene) at ppb level concentrations, with various ethanol backgrounds (0.5 and 2 ppm) and gas humidities (30, 50 and 70 % RH). The gas sensors were operated in temperature cycled operation. For signal processing, linear discriminant analysis was performed using features extracted from the conductance signals during temperature variations as input data. Both WO_3 sensor layers showed high sensitivity and selectivity to naphthalene compared to the other target gases. Of the two layers, the one prepared at higher oxygen partial pressure showed higher sensitivity and stability resulting in better discrimination of the gases and of different naphthalene concentrations. Naphthalene at concentrations down to 1 ppb could be detected with high reliability, even in an ethanol background of up to 2 ppm. The sensors show only low response to ethanol, which can be compensated reliably during the signal processing. Quantification of ppb level naphthalene concentrations was also possible with a high success rate of more than 99 % as shown by leave-one-out cross validation.

SiC-FET Sensors for ppb Level VOC

Exploring the Gas Sensing Performance of Catalytic Metal/Metal Oxide 4H-SiC Field Effect Transistors

D. Puglisi, J. Eriksson, M. Andersson, J. Huotari, M. Bastuck, C. Bur, J. Lappalainen, A. Schuetze, A. Lloyd Spetz

Materials Science Forum 858, 997-1000, 2016, DOI: 10.4028/www.scientific.net/MSF.858.997.

Abstract: Gas sensitive metal/metal-oxide field effect transistors based on silicon carbide were used to study the sensor response to benzene (C_6H_6) at the low parts per billion (ppb) concentration range. A combination of iridium and tungsten trioxide was used to develop the sensing layer. High sensitivity to 10 ppb C_6H_6 was demonstrated during several repeated measurements at a constant temperature from 180 to 300 °C. The sensor performance were studied also as a function of the electrical operating point of the device, i.e., linear, onset of saturation, and saturation mode. Measurements performed in saturation mode gave a sensor response up to 52 % higher than those performed in linear mode.

Integrated Sensor Systems

Integrated pre-concentrator gas sensor microsystem for ppb level benzene detection

M. Leidinger, M. Rieger, T. Sauerwald, C. Alépée, A.Schütze

Sensors & Actuators B: Chemical 236, 988-996, 2016, Doi:10.1016/j.snb.2016.04.064.

Abstract: An integrated microsystem for (indoor) air quality monitoring applications is presented. By combining a gas pre-concentrator based on metal-organic frameworks (MOFs) and a metal oxide semiconductor gas sensor, a device for detecting ppb levels of volatile organic compounds was designed, integrated and tested with benzene as a target gas. Two metal organic frameworks have been characterized for their suitability as gas pre-concentration materials for benzene and toluene using inverse gas chromatography and mass spectrometry measurements in order to obtain breakthrough values for gas adsorption. Both MOFs showed a higher pre-concentration effect compared to Tenax® TA, a state-of-the-art commercial adsorbent material. By depositing a MOF material on a micro hotplate integrated in a package together with a gas sensor, an integrated gas sensor microsystem was realized. This system has been characterized in FEM simulations concerning its gas pre-concentration capabilities and behavior. Test measurements were performed using benzene at concentrations of 10–1000 ppb. Both the simulations and the measurements show the suitability of the system design for the task. Significantly increased gas concentrations have been observed during thermal desorption from the pre-concentrator after gas adsorption at a low temperature.

Data Evaluation

Exploring the selectivity of WO_3 with Iridium catalyst in an ethanol/naphthalene mixture using multivariate statistics

M. Bastuck, D. Puglisi, J. Huotari, T. Sauerwald, J. Lappalainen, A. Lloyd Spetz, M. Andersson, A. Schütze

Thin Solid Films Special Issue SGS, 2015, doi:10.1016/j.tsf.2016.08.002.

Abstract: Temperature cycled operation and multivariate statistics have been used to compare the selectivity of two gate (i.e. sensitive) materials for gas-sensitive, silicon carbide based field effect transistors towards naphthalene and ethanol in different mixtures of the two substances. Both gates have a silicon dioxide (SiO_2) insulation layer and a porous iridium (Ir) electrode. One of it has also a dense tungsten trioxide (WO_3) interlayer between Ir and SiO_2. Both static and transient characteristics play an important role and can contribute to improve the sensitivity and selectivity of the gas sensor. The Ir/SiO_2 is strongly influenced by changes in ethanol concentration, and is,

thus, able to quantify ethanol in a range between 0 and 5 ppm with a precision of 500 ppb, independently of the naphthalene concentrations applied in this investigation. On the other hand, this sensitivity to ethanol reduces its selectivity towards naphthalene, whereas Ir/WO_3/SiO_2 shows an almost binary response to ethanol. Hence, the latter has a better selectivity towards naphthalene and can quantify legally relevant concentrations down to 5 ppb with a precision of 2.5 ppb, independently of a changing ethanol background between 0 and 5 ppm.

Calibration

A novel approach towards calibrated measurement of trace gases using metal oxide semiconductor sensors

C. Schultealbert, T. Baur, A. Schütze, S. Böttcher, T. Sauerwald

Sensors and Actuators B: Chemical 239, 390–396, doi:10.1016/j.snb.2016.08.002

Abstract: We present a method for quantitative measurements of metal oxide semiconductor gas sensors (MOS) which is based on relaxation of surface states using temperature cycled operation (TCO).

The method provides sensor response to toluene in form of a power law and a reliable quantification in the concentration range of about 10 ppb to 10 ppm. For the calibration of the sensor, a method is developed based on the equilibrium vapour headspace over a liquid solution of toluene and squalane yielding reliable concentrations in the range of 10 ppb to 600 ppm.

The calibration method was shown to be stable under ambient conditions and thus to be applicable for field calibration of gas sensors.

SENSIndoor Clip

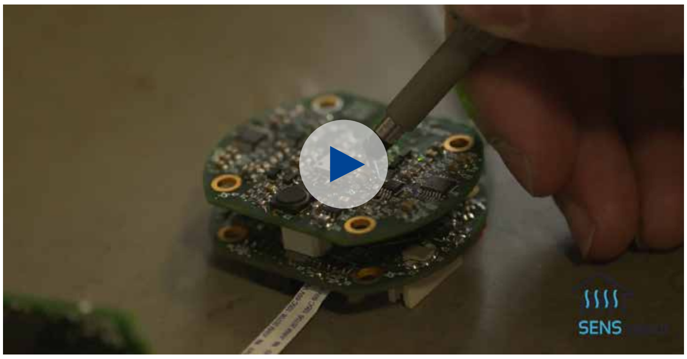
It’s done! We are happy to announce the release of our SENSIndoor clip.

As SENSIndoor is slowly coming to an end, we decided to produce a film clip to shed light on the past three years illustrating some of the steps taken towards the development of a smart sensor system to measure indoor air quality.

The clip highlights aspects of the work achieved in the project and introduces some of the partners involved. Animations have been added to explain the functionality and packaging process of the sensor system. Besides the illustration of the sensor system, the film strongly emphasises the mass production capability and market potential of the targeted product.

As an outlook into the future, further fields of application such as outdoor air quality control are described. The clip has been produced with the aim of raising awareness on the project impact for society and scientific community.

The film is available on the SENSIndoor website: www.sensindoor.eu/film



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M. Leidinger, T. Sauerwald, A. Schütze, C. Alépée, M. Rieger: **Integrated pre-concentrator gas sensor system for improved trace gas sensing performance.** IEEE Sensors 2016, Orlando, FL, USA, Oct. 30 - Nov. 2, 2016

M. Leidinger, T. Sauerwald, C. Alépée, A. Schütze: **Miniaturized integrated gas sensor systems combining metal oxide gas sensors and pre-concentrators.** EUROSENSORS XXX, Budapest, Hungary, September 4-7, 2016

I. Wilhelm, M. Rieger, J. Hürttlen, M. Wittek, C. Alépée, M. Leidinger, T. Sauerwald: **Novel low-cost selective pre-concentrators based on metal organic frameworks.** EUROSENSORS XXX, Budapest, Hungary, September 4-7, 2016

M. Andersson, M. Bastuck, J. Huotari, A. Lloyd Spetz, J. Lappalainen, A. Schütze, D. Puglisi: **SiC-FET sensors for selective and quantitative detection of VOCs down to ppb level.** EUROSENSORS XXX, Budapest, Hungary, September 4-7, 2016

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J. Huotari, T. Haapalainen, T. Baur, C. Alépée, J. Puustinen, J. Lappalainen: **Pulsed Laser Deposited Platinum Decorated Tin Oxide Nanotree Layers as highly Sensitive Gas Sensing Material.** CIMTEC 2016, 5th International Conference „Smart and Multifunctional Materials, Structures and Systems“, Perugia, Italy, June 5-9, 2016

D. Puglisi, M. Bastuck, M. Andersson, A. Lloyd Spetz, A. Schütze, J. Huotari, J. Lappalainen, V. Kekkonen, J. Liimatainen: **Functional Oxide Materials for high Performance SiC-FET Sensors for Indoor Air Quality Contro.** CIMTEC 2016, 5th International Conference „Smart and Multifunctional Materials, Structures and Systems“, Perugia, Italy, June 5-9, 2016

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THANK YOU!!!

Working together in a multinational, diverse and passionate team was an inspiring experience for all of us. We would like to thank everybody who contributed to three years of ambitious project work within SENSIndoor and helped to make the project a success.

SENSIndoor Fact Sheet

Full Title
Nanotechnology-based intelligent multi sensor system with selective pre-concentration for indoor air quality control

Programme
7th Framework Programme of the European Commission – NMP – Small or Medium-sized Collaborative Projects

Duration
36 months (start date: January 2014)

Project Funding
3.399.995,00 €

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This issue was published in November 2016.

Disclaimer

The SENSIndoor project (Project Identifier: 604311) is funded by the European Commission under the Seventh Framework Programme.

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