European Network on New Sensing Technologies for Air Pollution Control and Environmental Sustainability - *EuNetAir*

COST Action TD1105

Final Meeting at PRAGUE (CZ), 5-7 October 2016

New Sensing Technologies for Air Quality Monitoring

Action Start date: 01/07/2012 - Action End date - EXTENSION: 15/11/2016

The SENSIndoor FP7 Project: Main Results, Lessons Learned and Outlook

UNIVERSITÄT DES SAARLANDES

Lehrstuhl für LMT Messtechnik

Andreas Schütze

WG2 leader

Lab for Measurement Technology, Saarland University, Germany

schuetze@LMT.uni-saarland.de

COST EUROPEAN COOPERATION IN SCIENCE AND TECHNOLOGY

EuNetAir

European Network on New Sensing Technologies for Air-Pollution Control and Environmental Sustainability

COST is supported by the EU Framework Programme

European Science Foundation ESF provides the COST Office through a European Commission contract

Nanotechnology based intelligent multi-**SEN**sor **S**ystem
with selective pre-concentration for **Indoor** air quality control

SENSIndoor

Integrated microsensor system with selective pre-concentration for ubiquitous IAQ monitoring

Indoor Air 2016
July 7, 2016 - Ghent, Belgium

Andreas Schütze
Saarland University, Lab for Measurement Technology

LMT · UNIVERSITÄT DES SAARLANDES · SEVENTH FRAMEWORK PROGRAMME

VOCs: key for Indoor Air Quality

- Volatile Organic Compounds (VOCs) are highly relevant for IAQ
- Some are proven or suspected to be carcinogenic
- Resulting target concentrations are low ppb or even sub-ppb

  → **High sensitivity required**
- Benign VOCs (e.g. ethanol) can occur at much higher conc. (ppm)

  → **High selectivity required**
- Most relevant target VOCs according to European studies: formaldehyde, benzene, naphthalene

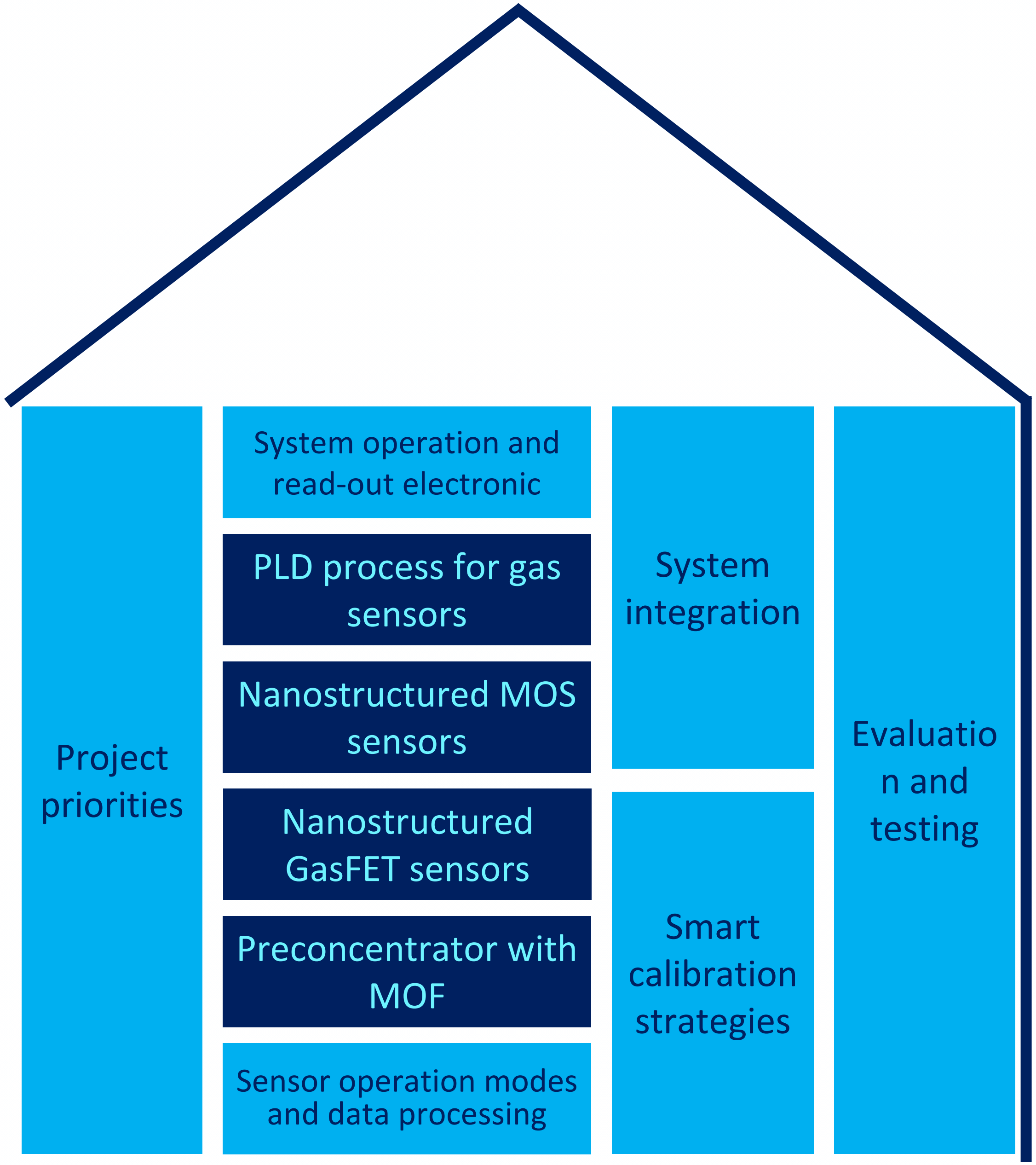
Target gas	Guideline values	
	µg/m³	**ppb**
Formaldehyde [1]	100	81.3
Benzene [2]	5	1.57
Naphthalene [1]	10	1.9

[1]: WHO guidelines for indoor air quality (2010)
[2]: Umweltbundesamt Infoblatt Benzol (12/2010)
Note: some national regulations target even lower concentration limits, e.g. France

The consortium – a great team

Universität des Saarlandes	Universität des Saarlandes, Germany Department of Mechatronics Laboratory for Measurement Technology *Prof. Andreas Schütze*
Linköping University	Linköping University, Sweden Department of Physics, Chemistry and Biology Division of Applied Sensor Science *Prof. Anita Lloyd Spetz*
University of Oulu	University of Oulu, Finland Functional Electroceramics Thin Film Group *Prof. Jyrki Lappainen*
Fraunhofer ICT	Fraunhofer Gesellschaft zur Förderung der angewandten Forschung e.V., Germany Fraunhofer Institute for Chemical Technology *Dr. Jürgen Hürttlen*
Picodeon	Picodeon Ltd. Oy, Finland *Jari Liimatainen*
SGX Sensortech	SGX Sensortech S.A., Switzerland *Dr. Christine Alépée*
SENSIC Clean air sensors	SenSiC AB, Sweden *Dr. Mike Andersson*
3S Gas sensing solutions	3S – Sensors, Signal processing, Systems GmbH, Germany *Thorsten Conrad*
NanoSense	NanoSense S.à.r.l., France *Olivier Martimort*
EURICE	European Research and Project Office GmbH (Eurice), Germany *Corinna Hahn*

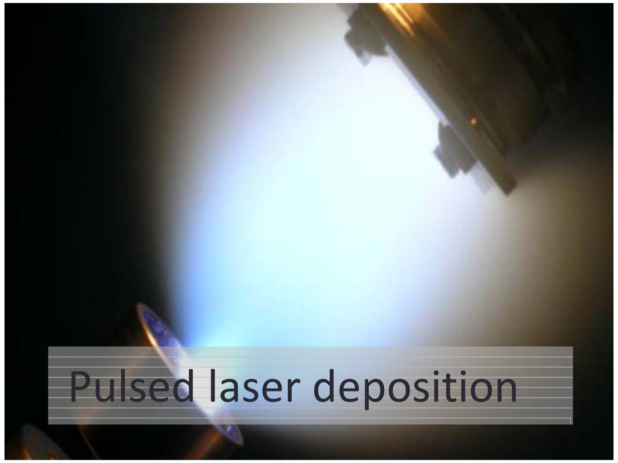
SENSIndoor develops a pulsed laser deposition (PLD) process for gas sensors

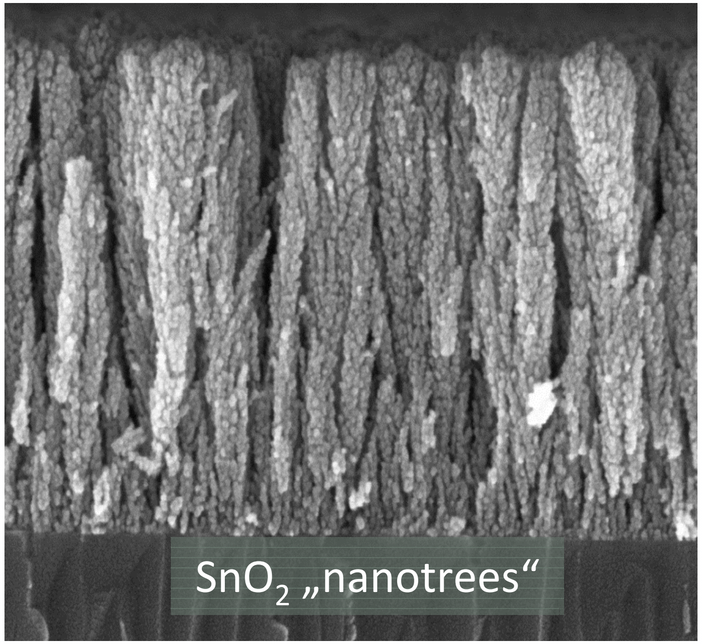


PLD process for gas sensors

SENSIndoor

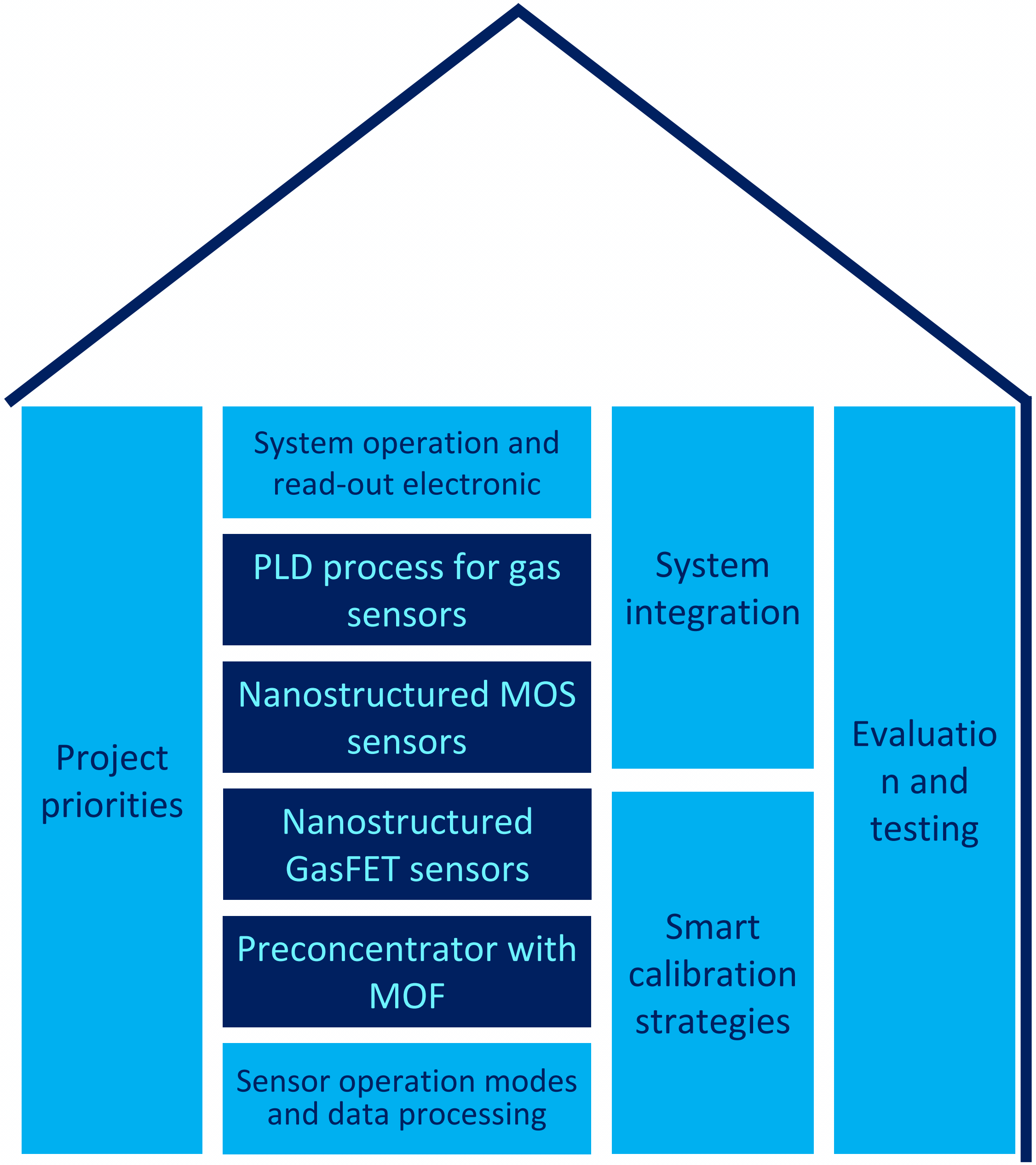
- Well suited deposition method for porous nanocrystalline layers of SnO_2 and WO_3 (for MOS sensors) and for dense layers with noble metal decoration (for Gas-FET sensors)
- Noble metal doping with alternating deposition
- Suitable for wafer level deposition: process scale-up demonstrated
- Partners: Univ. of Oulu, Picodeon





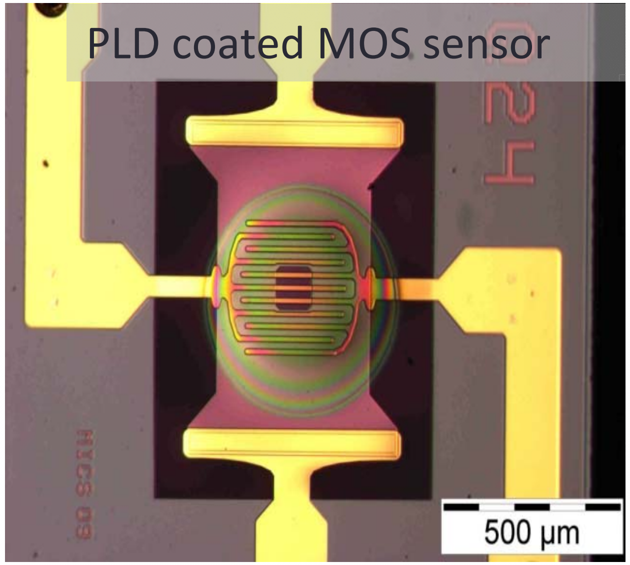
J. Huotari, V. Kekkonen, T. Haapalainen, M. Leidinger, T. Sauerwald, J. Puustinen, J. Liimatainen, J. Lappalainen, *Pulsed laser deposition of metal oxide nanostructures for highly sensitive gas sensor applications*, Sensors & Actuators B (2016), accepted

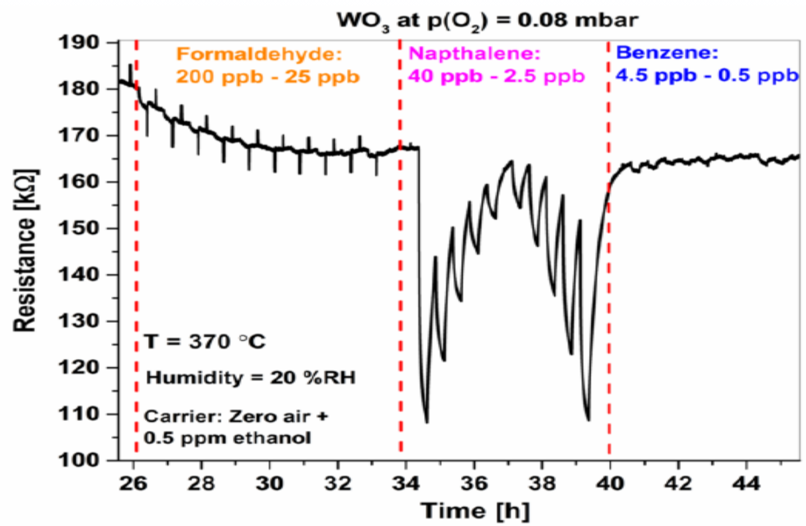
SENSIndoor develops new nanostructured metal oxide semiconductor (MOS) sensors



Nanostructured MOS sensors

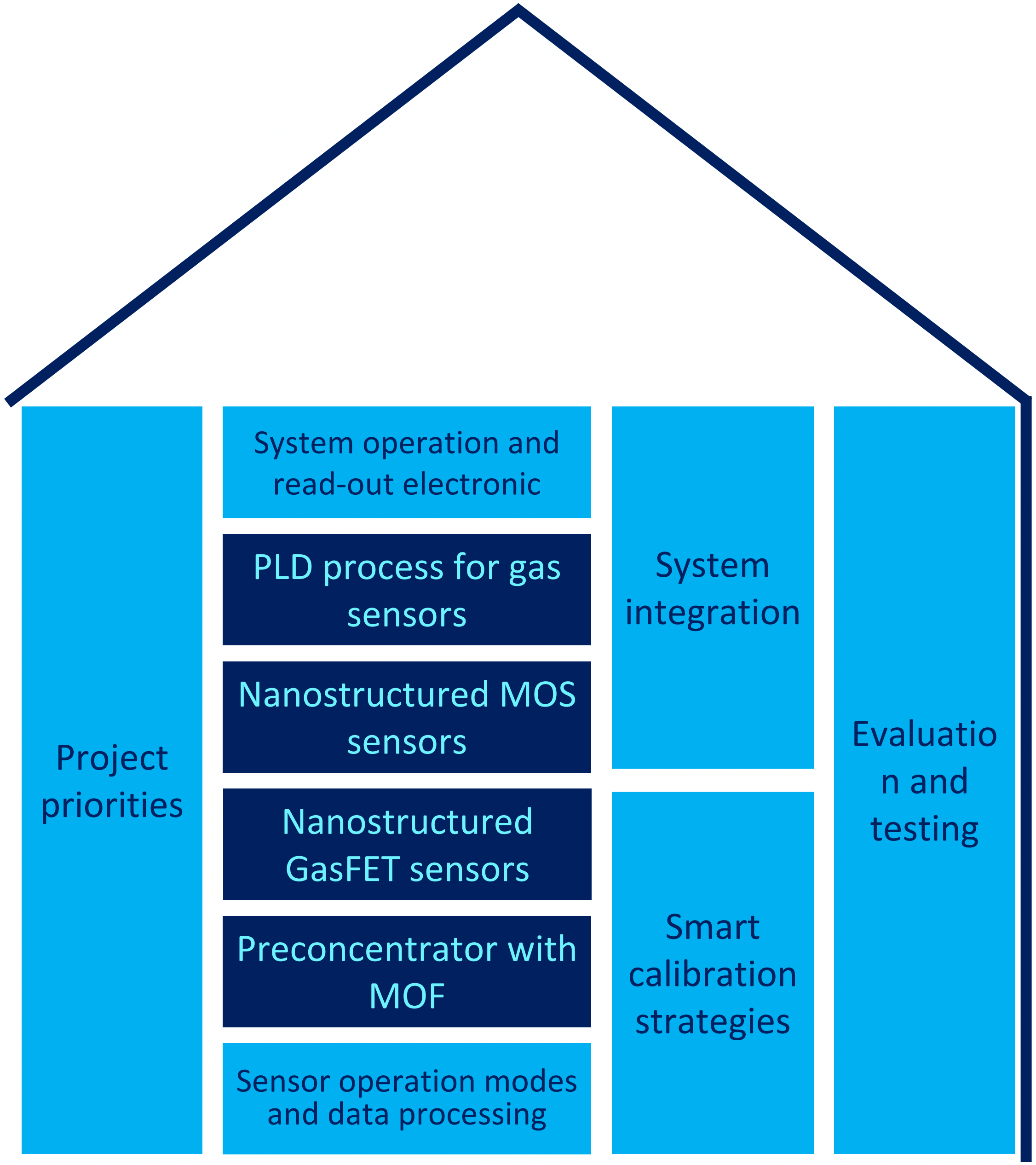
- Optimized micro-hotplates
- MOS sensors with PLD coated layers show high sensitivity in the ppb range
- Various layer types (SnO_2, WO_3) with and without noble metal catalyst in order to enhance selectivity
- Partners: SGX, Univ. of Oulu





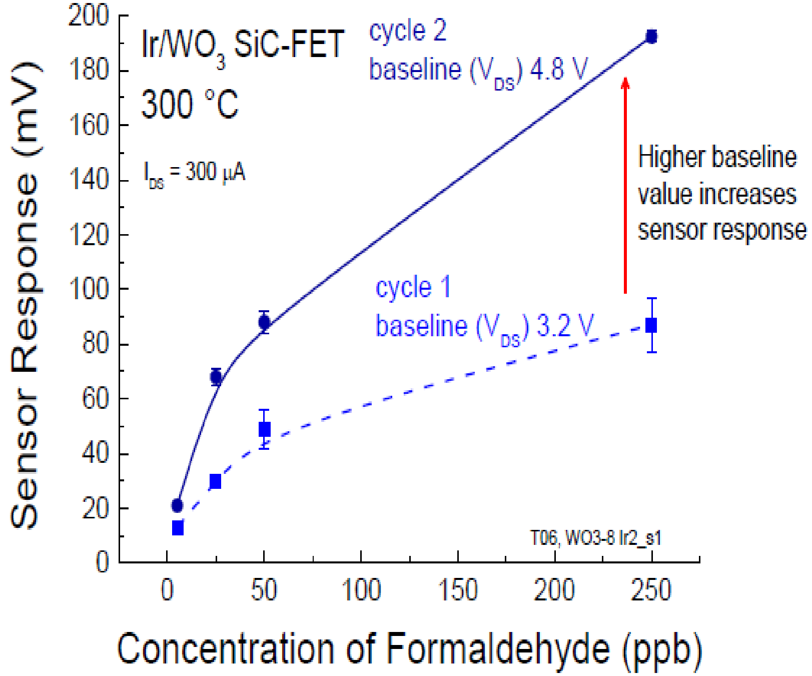
M. Leidinger, J. Huotari, T. Sauerwald, J. Lappalainen, A. Schütze: Selective detection of naphthalene with nanostructured WO_3 gas sensors prepared by pulsed laser deposition
J. Sens. Sens. Syst. (2016), 5, 147-156, doi: 10.5194/jsss-5-147-2016

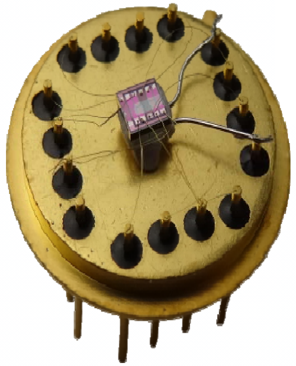
SENSIndoor develops new gas sensitive SiC field effect transistors (SiC-FET) with nanostructured gate materials



Nanostructured SiC-FET sensors

- Optimized platforms w integrated heater
- Metal oxide (WO_3) with Ir as gate material
- High response in the ppb range, especially vs. formaldehyde
- Additional contacts with metal electrodes to increase the stability
- Partners: LiU, SenSiC

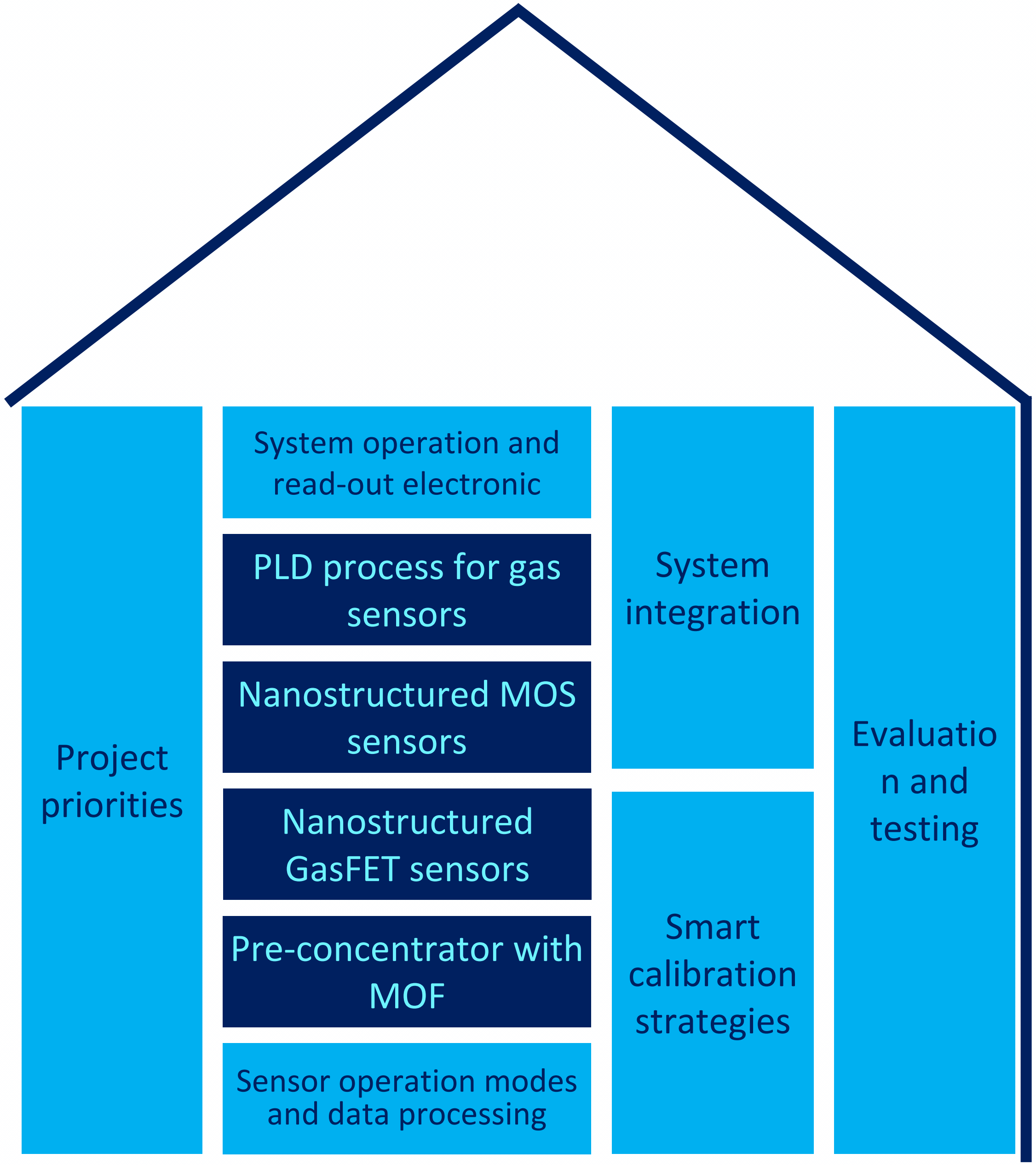




GasFET sample with WO_3 + Ir gate

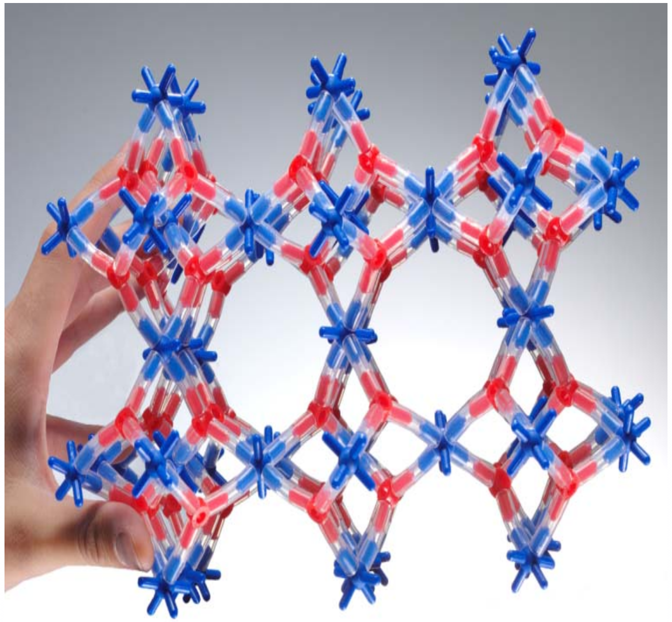
SENSIndoor

SENSIndoor has developed a novel pre-concentrator concept based on metal organic frameworks (MOF)



Metal organic frameworks (MOF)

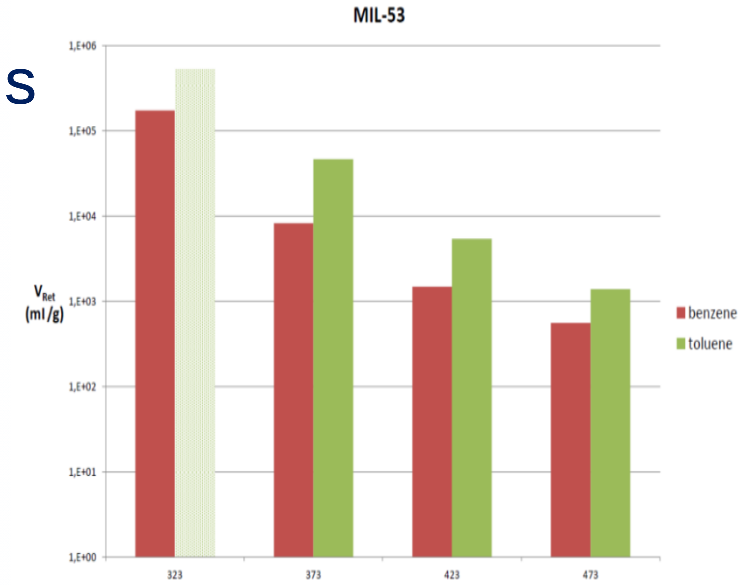
- Crystalline materials composed of metal centers & organic linkers
- Ultrahigh porosity
- Extremely large internal surface areas (up to several 1000 m^2/g)
- Tunable chemical and physical properties
- Partner: Fraunhofer ICT



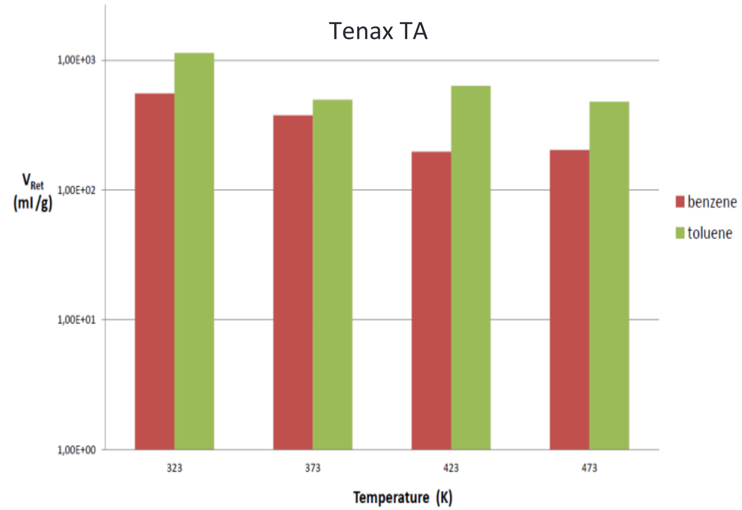
MOF pre-concentrators

- Pre-concentrators based on MOF shows two orders of magnitude higher sorption efficiency than conventional materials (e.g. Tenax TA)
- Efficient sampling of benzene to boost sensitivity and selectivity
- Partners: FhG-ICT, SGX, USAAR-LMT

M. Leidinger, M. Rieger, T. Sauerwald, M. Nägele, J. Hürttlen, A. Schütze: Trace gas VOC detection using metal-organic frameworks micro pre-concentrators and semiconductor gas sensors
EUROSENSORS 2015, Freiburg, Germany, September 6 to 9, 2015,
doi: 10.1016/j.proeng.2015.08.719

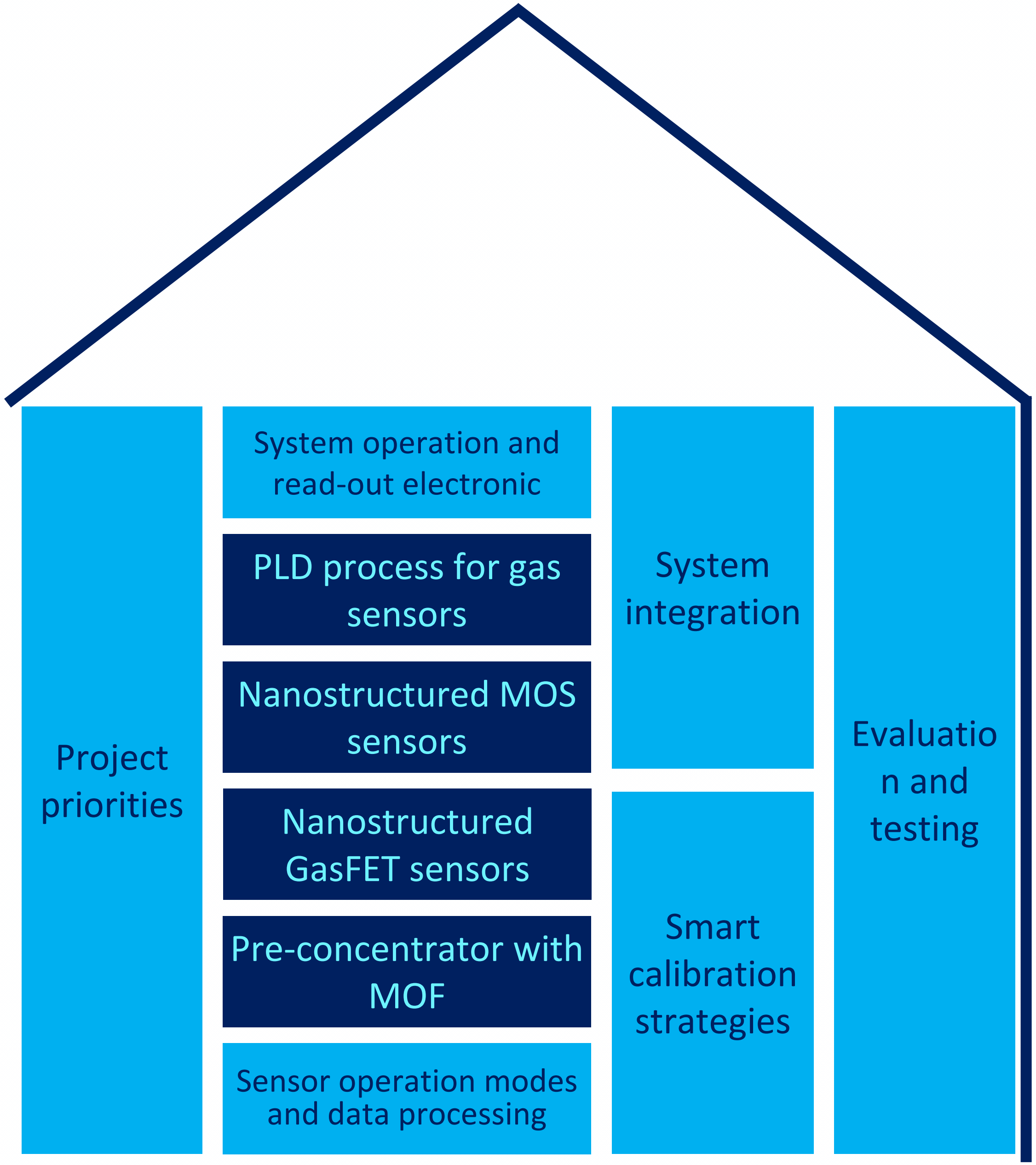


Breakthrough value in iGC to determine the partition coefficient



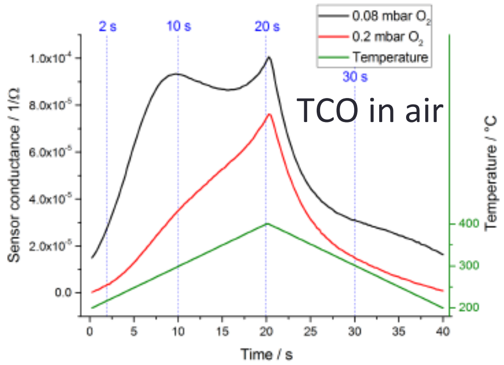
SENSIndoor

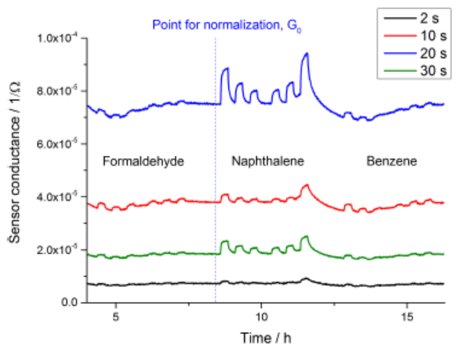
SENSIndoor uses smart operation modes for sensor and data processing for detection and quantification



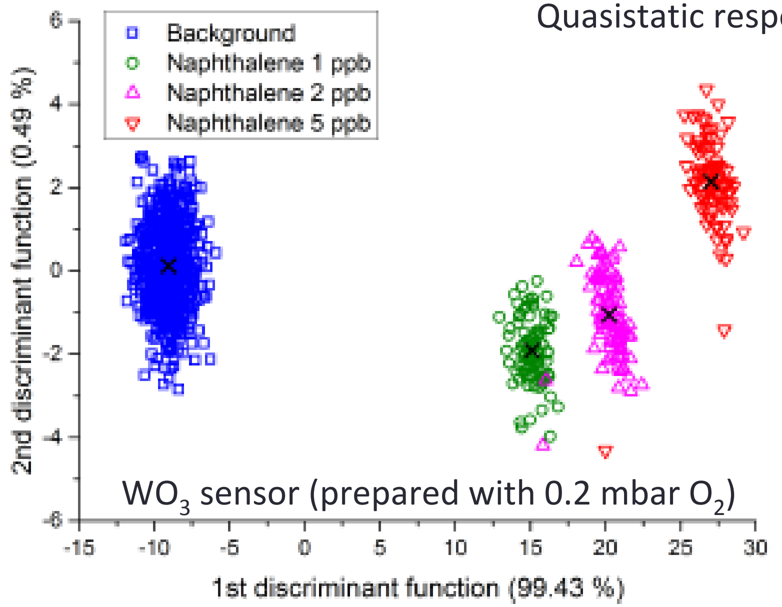
Smart operation modes and data processing

- Multiple signal generation by temperature cycled operation (TCO) and gate bias cycling (GBCO)
- Data processing for detection (e.g. LDA) and quantification (e.g. PLSR)
- Selective and quantitative VOC detection at ppb level
- Partners: USAAR-LMT, LiU



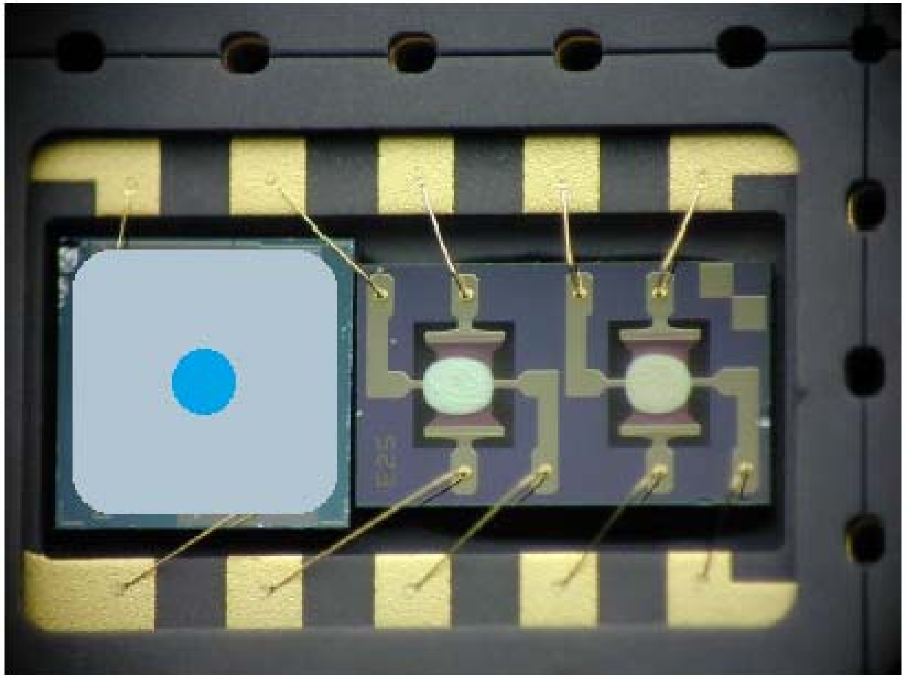


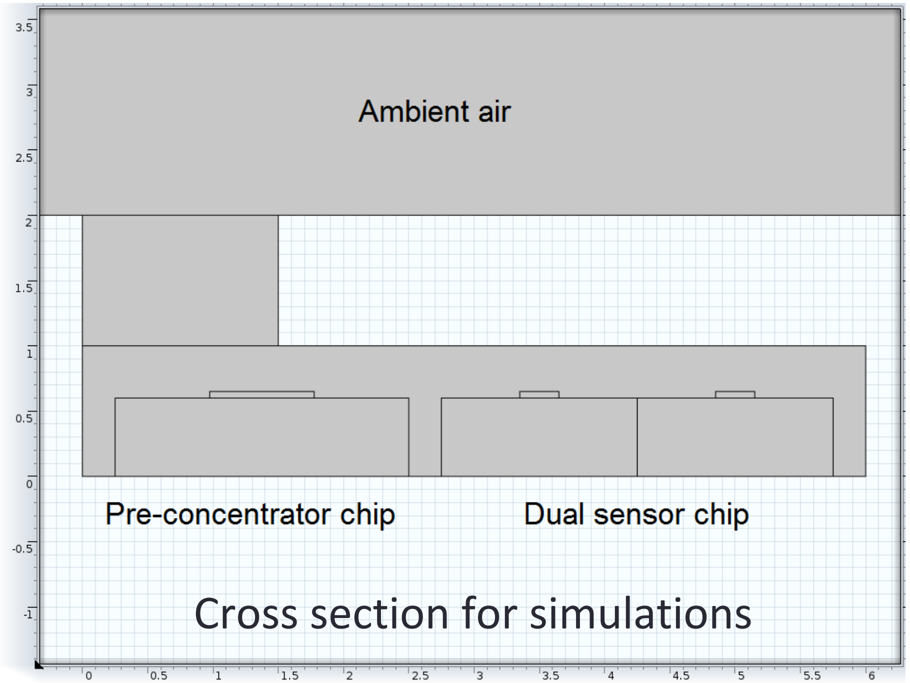
Quasistatic response



M. Leidinger, J. Huotari, T. Sauerwald, J. Lappalainen, A. Schütze:
Selective detection of naphthalene with nanostructured WO_3 gas sensors prepared by pulsed laser deposition J. Sens. Sens. Syst. (2016), 5, 147-156, doi: 10.5194/jsss-5-147-2016

Combination of MOS sensor with µPC





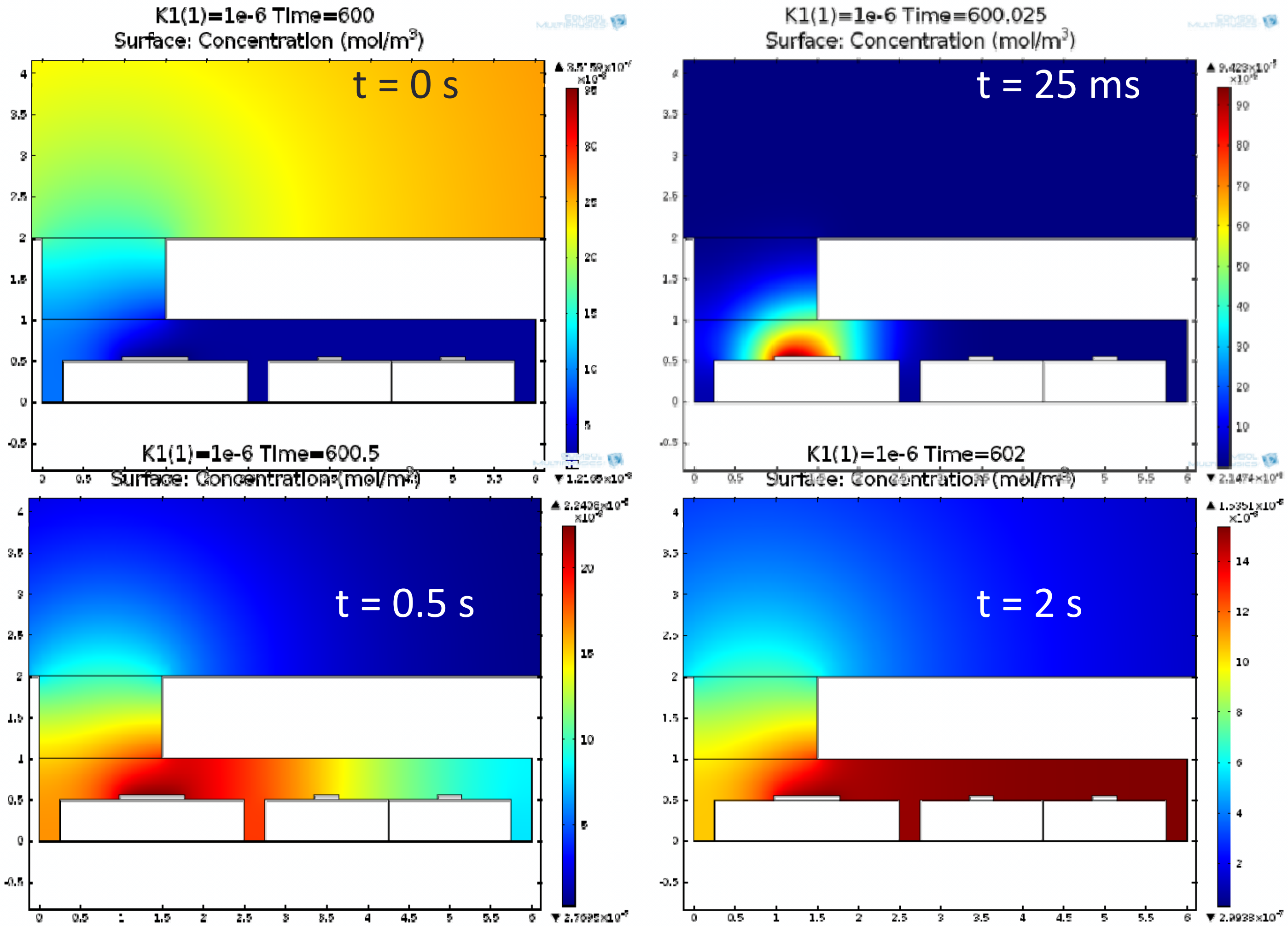
SMD ceramic package
(5x7 mm² footprint)

Lid with gas access
not shown

Left:
µPC chip
(SGX coated by FhG-ICT)
MOF material
Ø ≈ 300 µm

Right:
Dual gas sensor chip
(SGX Sensortech)
1x WO_3 undoped
1x WO_3 doped

Simulation of adsorption/desorption cycles (Comsol)



Background gas concentration: $4*10^{-7}$ mol/m³ (approx. 10 ppb)

Accumulation: 600 s

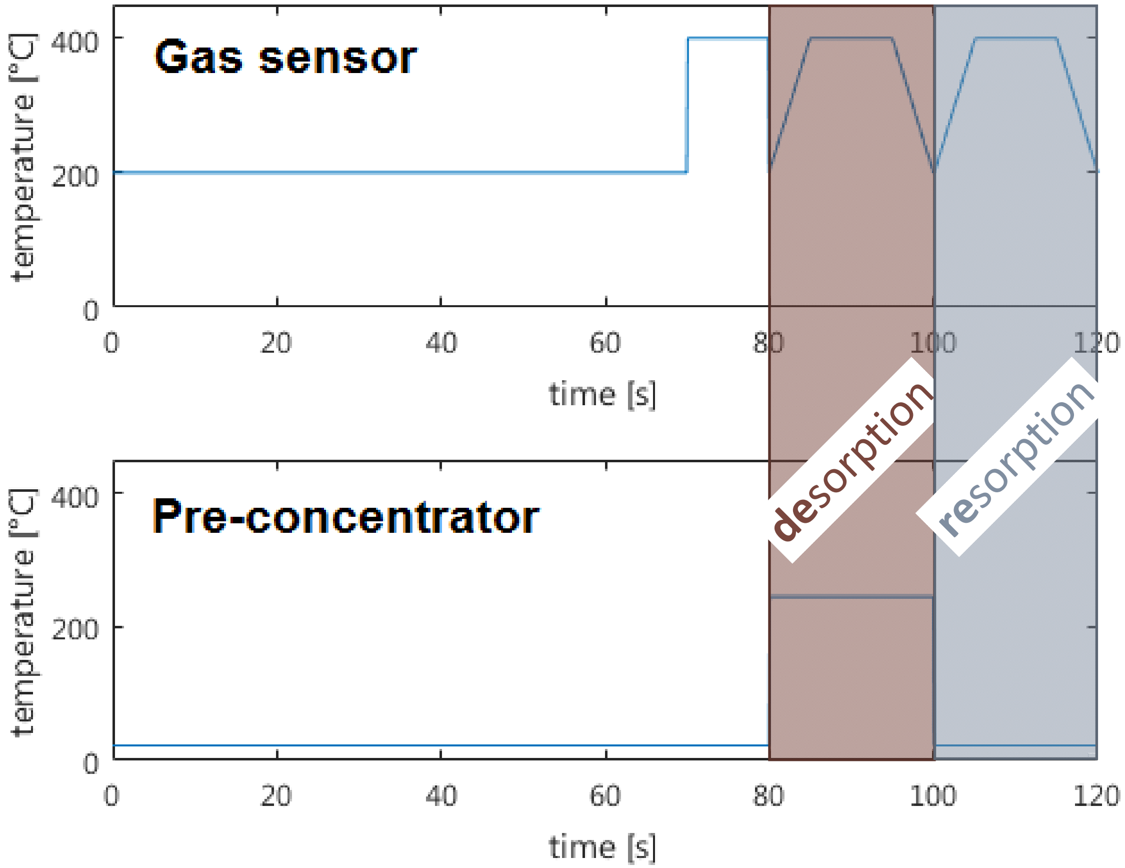
Desorption: 10 s

Simulations show approx. 2 orders of magnitude higher concentration

Note: colors show relative scales in each image

M. Leidinger, M. Rieger, T. Sauerwald, C. Alépée, A. Schütze: Integrated pre-concentrator gas sensor microsystem for ppb level benzene detection
Sensors and Actuators B (2016), doi: 10.1016/j.snb.2016.04.064

Operating mode for combined MOS sensor and µPC



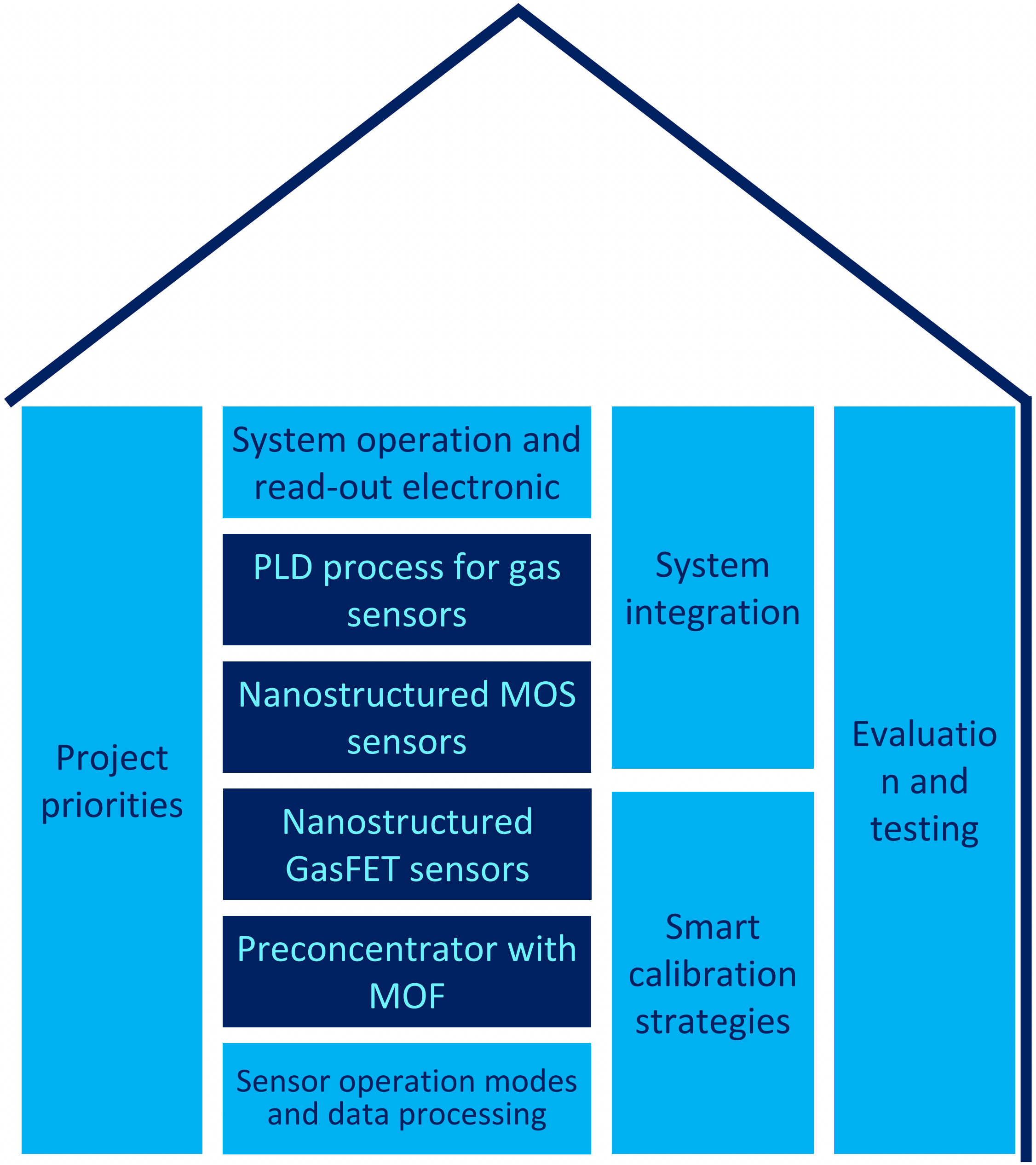
ramp: "broadband measurement" (instead of temp. step)

compare **de**sorption and **re**sorption cycle to obtain sensor response and achieve differential measurement for higher selectivity

Application specific optimization: sensitivity, selectivity and power consumption

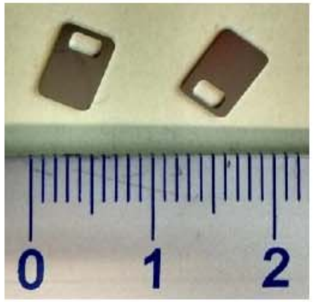
SENSIndoor

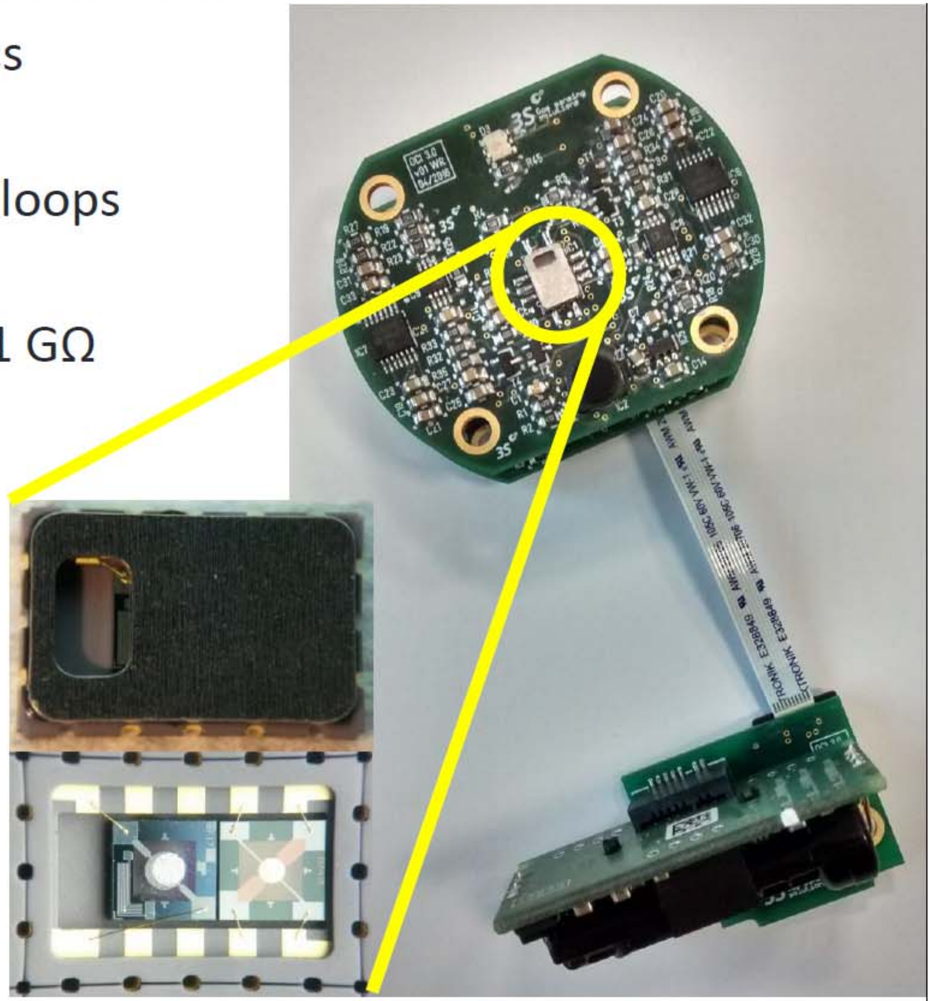
SENSIndoor integrates sensor and pre-concentrator with electronic readout and data processing



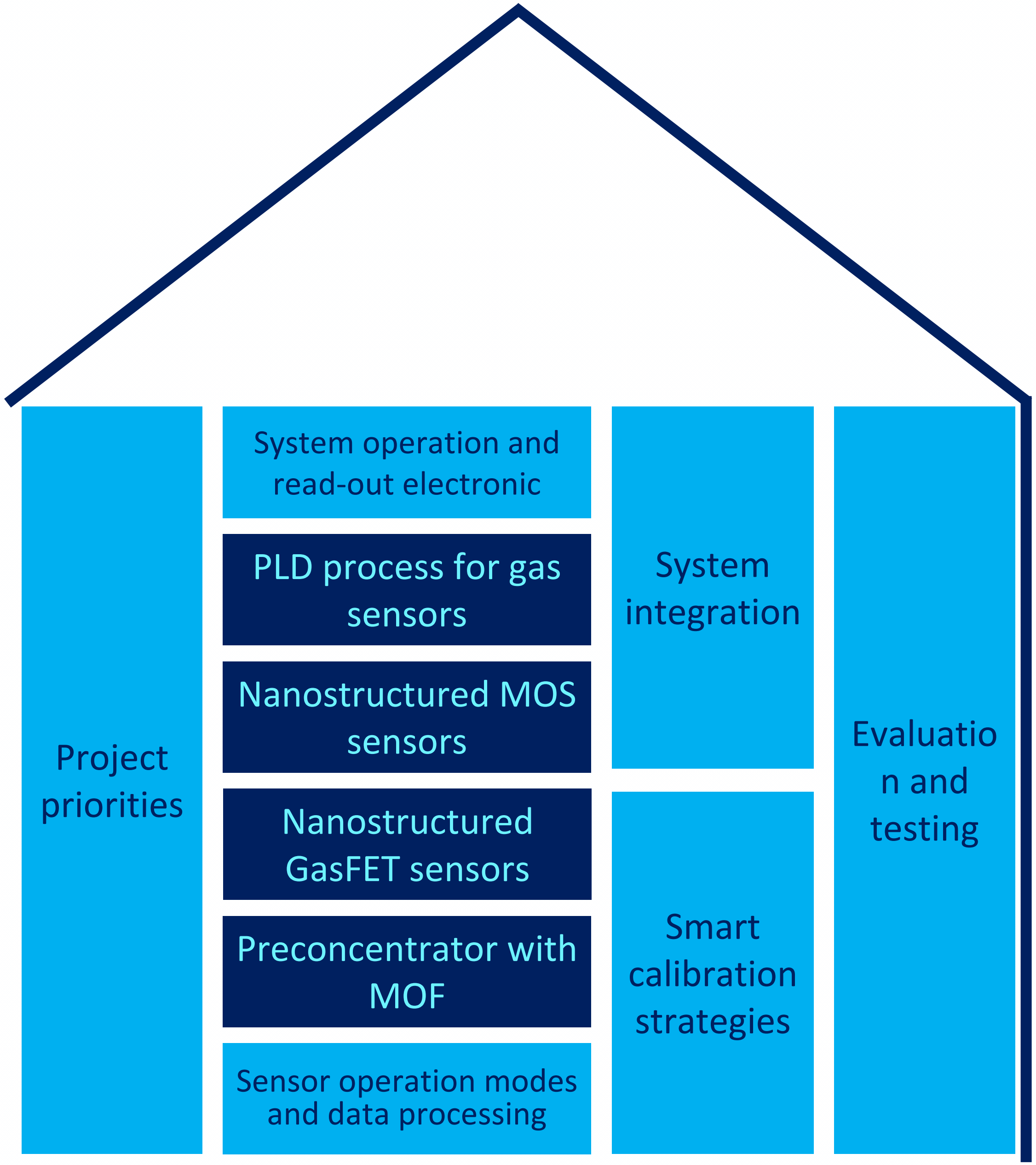
Sensor operation and read-out electronics

- Integration of pre-developed electronics
- 3 independently controlled, fully synchronized analog resistance control loops (1 PC + 2 MOS) 1% accuracy
- highly dynamic MOS read-out 250 Ω – 1 GΩ 300 digits / decade resolution
- CO_2 and µSD card extension
- small series production for SENSIndoor field tests
- Partners: 3S, NanoSense





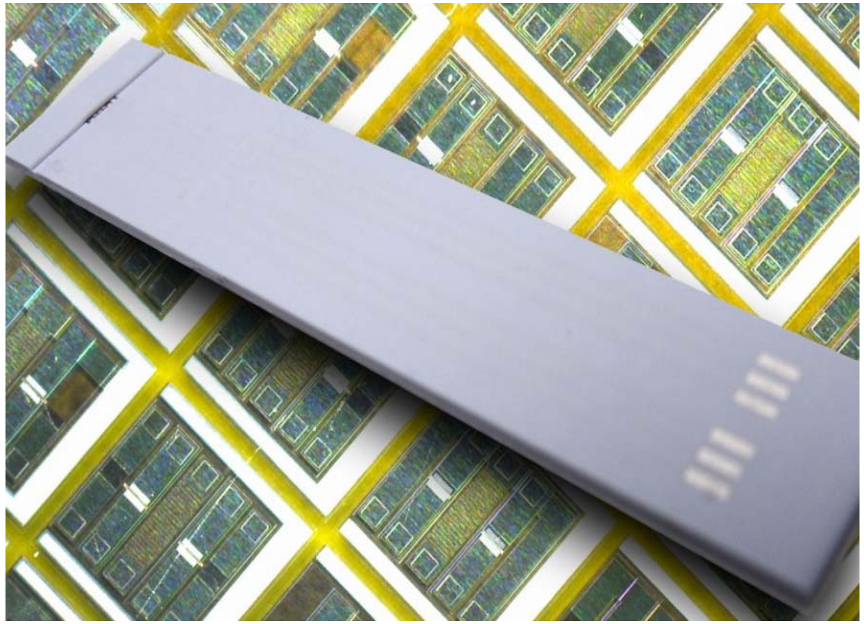
SENSIndoor integrates sensor and pre-concentrator with electronic readout and data processing



System integration: SiC-FET

New integration concept for SiC-FETs and SiC hotplates w LTCC package

- Partners: LiU, SenSiC, Univ. of Oulu



GasFET integration in a Low Temperature Co-fired Ceramic (LTCC) module

System integration: MOS w µPC

Simple integration concept for MOS sensors and micro-pre-concentrators – scale-up possible with proven technologies

- Partners:
  USAAR-LMT,
  FhG-ICT, SGX

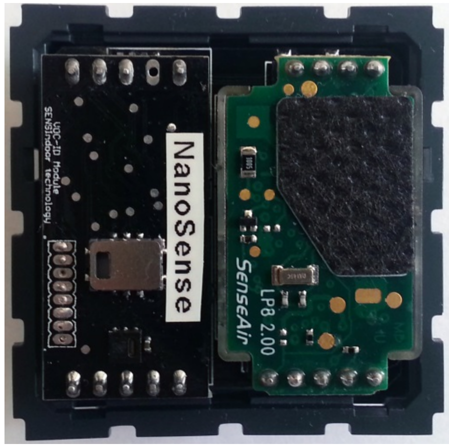
MOS and µPC integration in an SMD package with controlled gas access

Sensor system integration

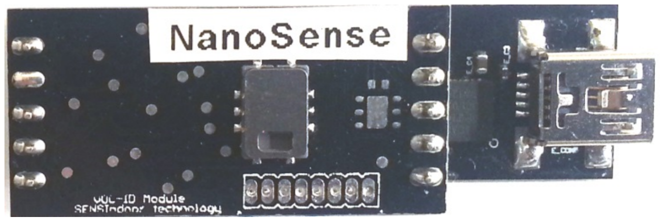
- Flush mount enabled design (50 x 40 mm) for unobtrusive installation
- µSD card and CO2 sensor extension for field test raw data collection
- Field calibration adapter attachable

Gas entry to
sensor/pre-concent

…cal visualisation
…tem state



Optimized for low BOM



(NanoSense)

Gas e…
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minimize
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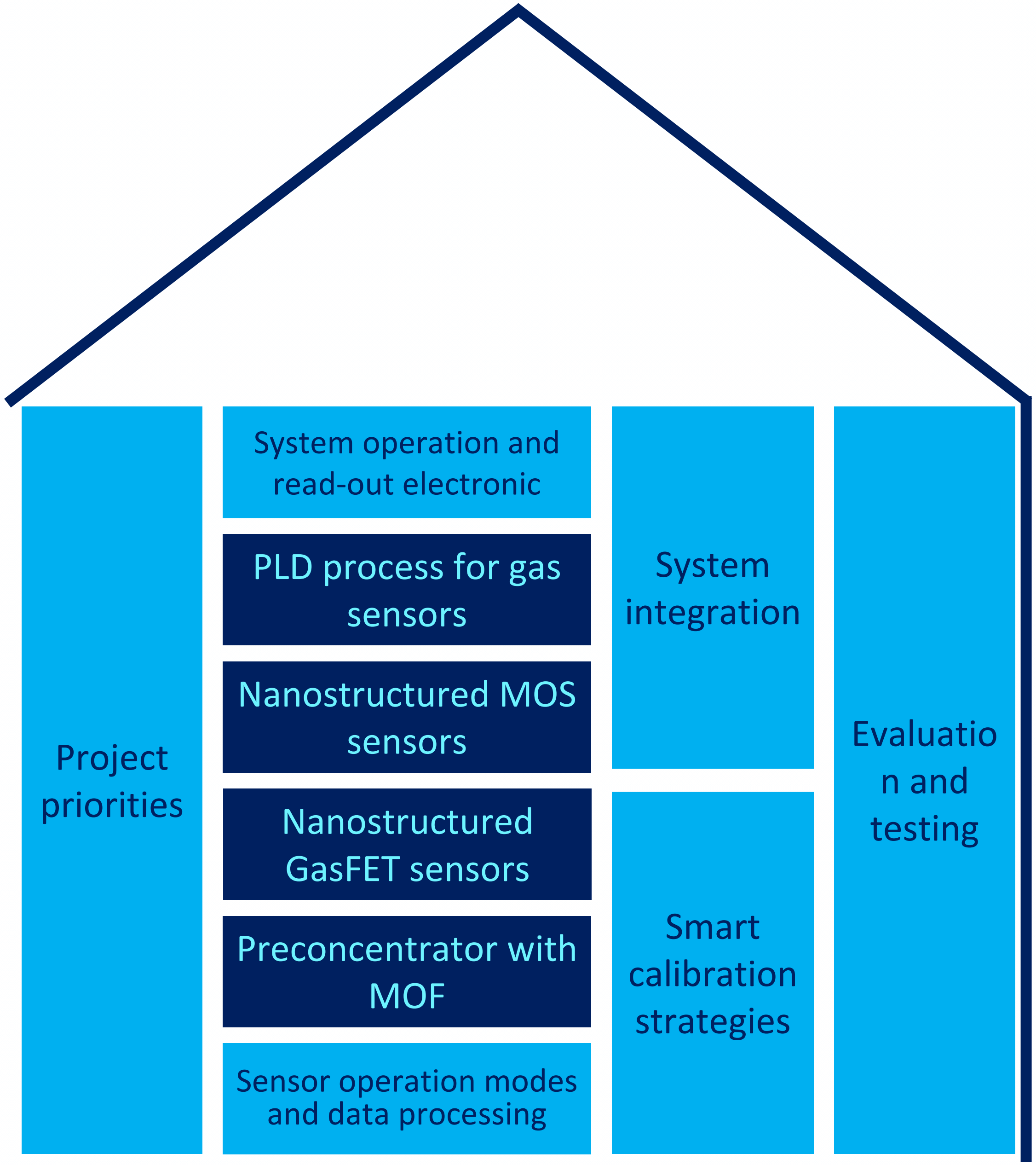
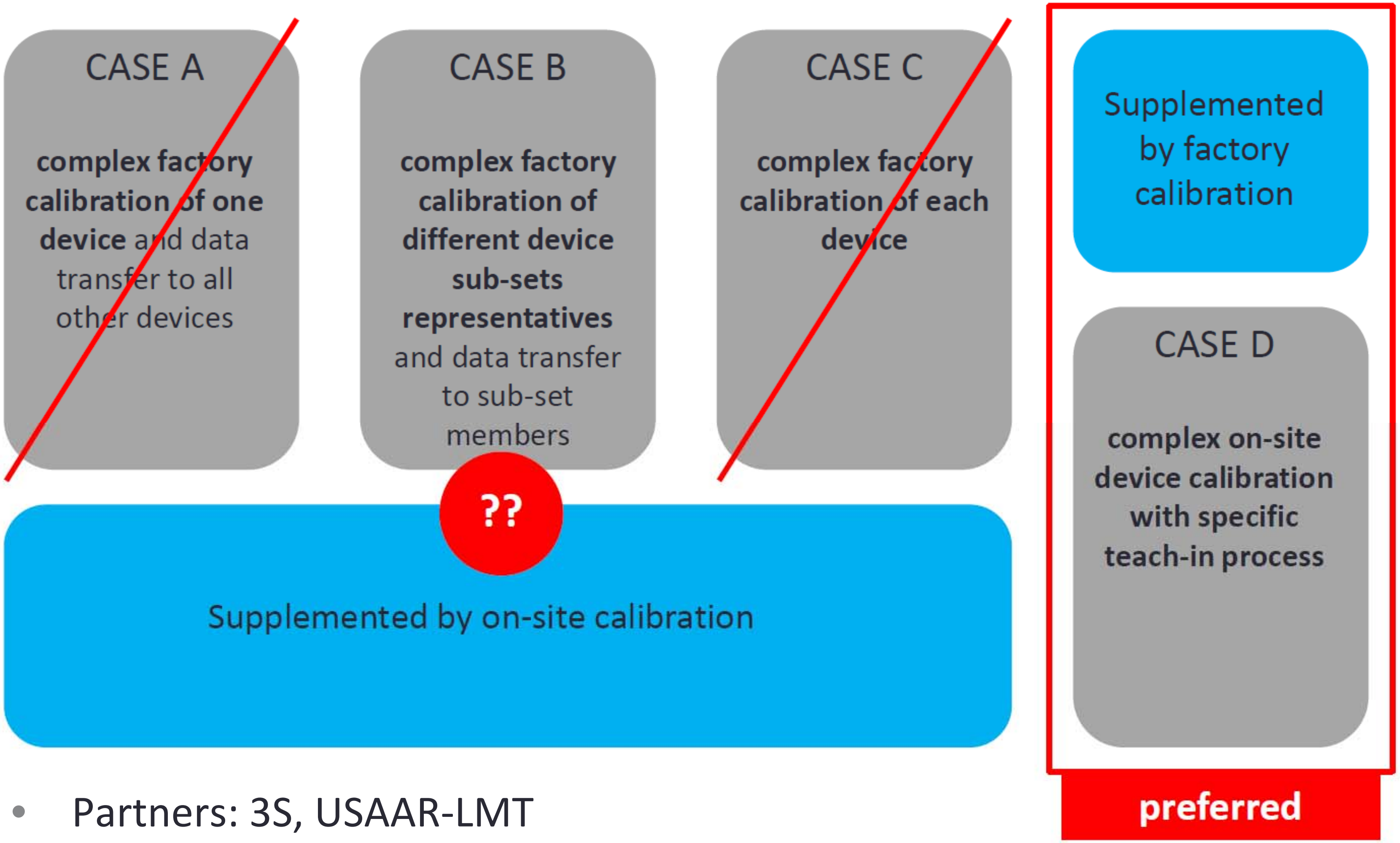
SENSIndoor integrates sensor and pre-concentrator with electronic readout and data processing



Smart calibration strategies



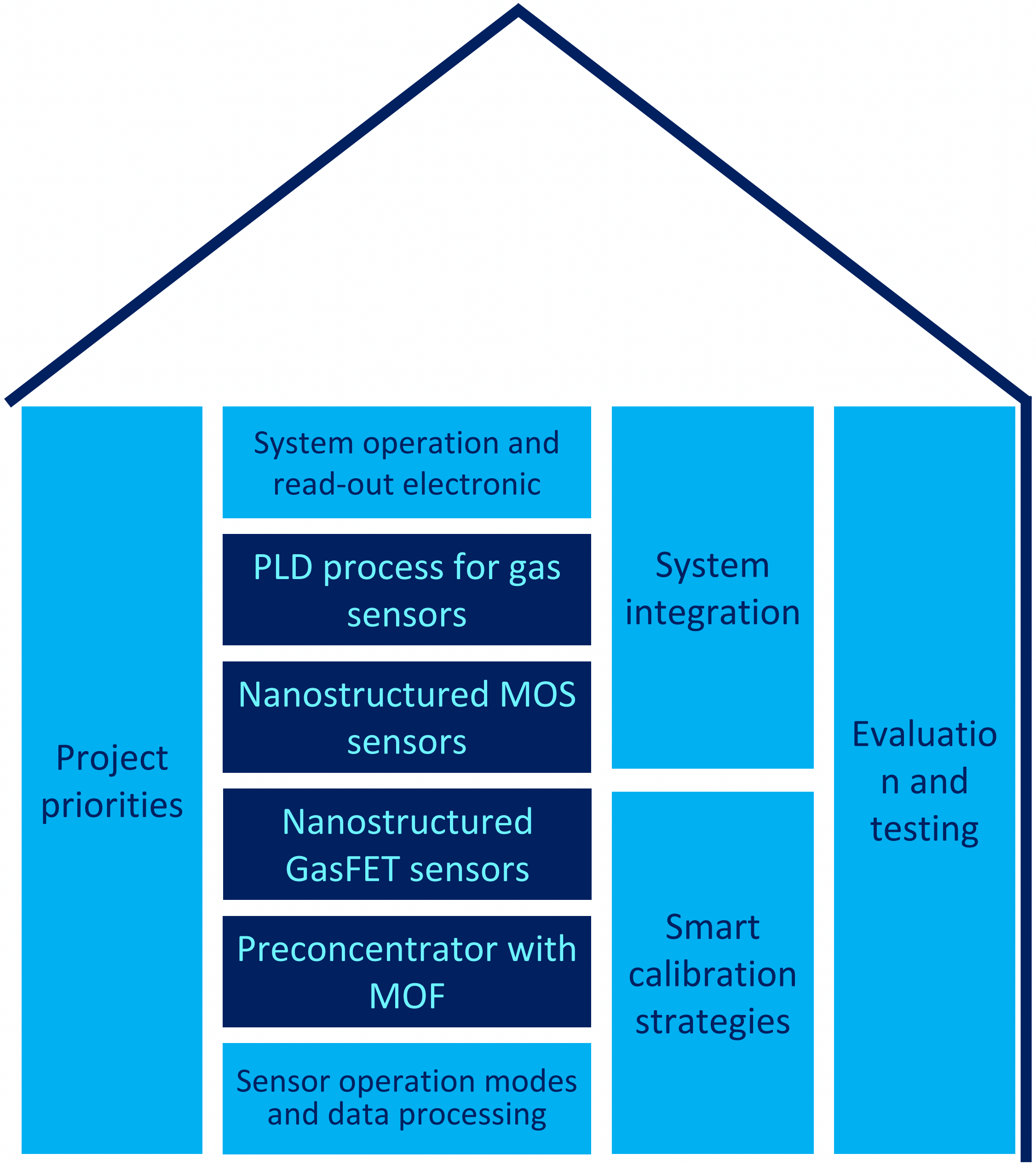
- Partners: 3S, USAAR-LMT

On-site calibration with simple and flexible approach

- Easy to use mobile calibration standard
- Exploiting the vapor pressure of (highly) diluted solutions (Henry law)
  - Successfully tested by dilution series with Toluene (C_7H_8) and Squalane ($C_{30}H_{62}$ - non volatile liquid)
  - Different dilution levels provide needed concentrations (also zero air)
- To Do:
  - Transferring results to target VOCs
  - Replacing liquid by a wax or gel

SENSIndoor

SENSIndoor integrates sensor and pre-concentrator with electronic readout and data processing



SENSIndoor

Upcoming

We cordially invite you to the
presentation of the final project results!

Public presentation at the SENSIndoor final meeting:
November 15th-16th, 2016
Saarbruecken, Germany
Day 1 (afternoon): System components and technology
Day 2 (morning): System integration and testing

More information available at:
http://www.sensindoor.eu/

Lessons learned – recommendations

- Step up: make use of available opportunities/calls!
- Focus: be clear on objectives and common targets!
- Team up: don´t underestimate the team spirit!
- Fix rules: especially for publications/patents!
- Set procedures: define process for reports, deliv., publications!
- Communicate: keep everyone in the loop!
- Disseminate: not only scientific pub., but press releases…!
- Join forces: with other projects, in networks etc.!
- Go out: independent evaluation helps dissemination!

SENSIndoor

Nanotechnology based intelligent multi-**SEN**sor **S**ystem
with selective pre-concentration for **Indoor** air quality control

Further information:
www.sensindoor.eu

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LMT Lehrstuhl für Messtechnik

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