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

## COST Action TD1105

## INTERNATIONAL WG1-WG4 MEETING on

***New Sensing Technologies and Methods for Air-Pollution Monitoring***

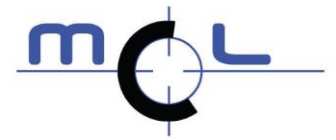
**European Environment Agency - EEA**

**Copenhagen, Denmark, 3 - 4 October 2013**

**Action Start date: 01/07/2012 - Action End date: 30/06/2016 - Year 2: 2013-2014 (*Ongoing Action*)**

# MSP - Multi Sensor Platform for Smart Building Management

**(FP7-ICT-2013-10 Collaborative Project, No. 611887)**




**Anton Köck**

**Materials Center Leoben, Austria**

COST is supported by the EU Framework Programme

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

[e-COST] New nomination in Action TD1105 - Windows Internet Explorer

https://exchange.mcl.at/owa/?ae=Item&a=Open&t=IPM.Note&id=RgAAAACebhKMQNgbQK2HeWKgJ9hjBwDYOzf55DoGS5%2f0xxghQ%2f5PA

Antworten Allen antworten Weiterleiten

# [e-COST] New nomination in Action TD1105

noreply@cost.eu

**An:** Anton Köck

Donnerstag, 26. September 2013 15:09

Dear Anton KOCK,

You have been nominated as a MC Member [TD1105 AT] to COST Action TD1105 by Dr MIELING (COST National Coordinator [AT]).

To complete your nomination, please follow the link below:
https://e-services.cost.eu/?module=user&action=activationCode&userParam[code]=NOMINATION_199db1e79d1866414333bbac3e4c952c

Note: Please do not log in to eCOST directly; you must follow the link above in order to complete your nomination.

After clicking on this link you will be prompted to either
- Login in to eCOST using your existing eCOST login details (if you already have an eCOST profile), or
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Please take a few moments to fill in (or update) your personal information in e-COST and do not forget to register your bank details in for any future reimbursement by COST.

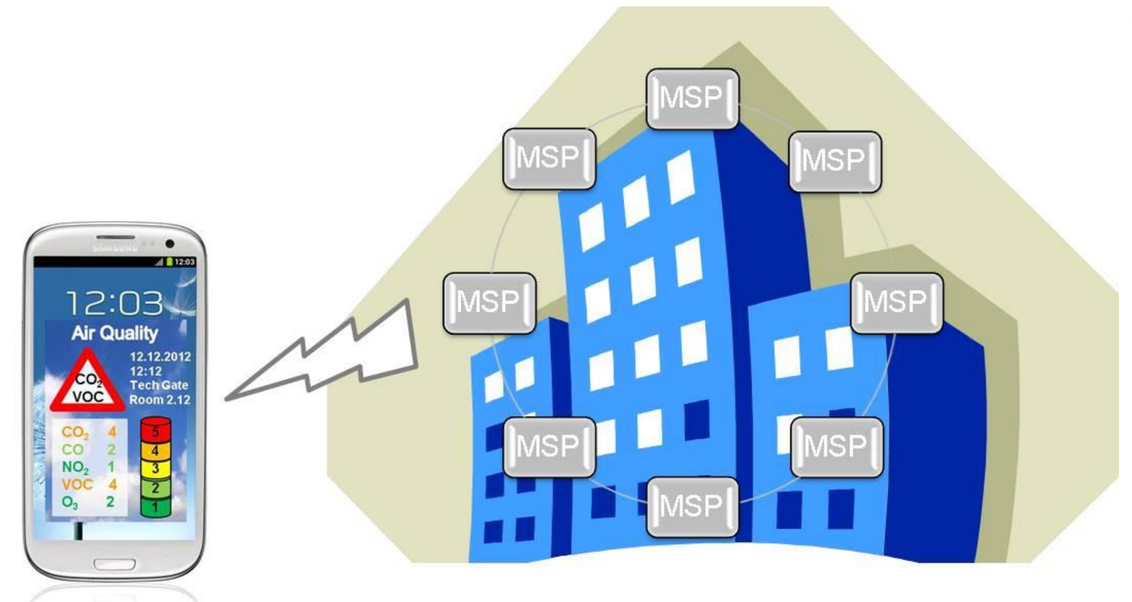
More information on the rules for reimbursement (Vademecum) and Rules and procedures for implementing COST Actions can be found on the following page:

100%

# MSP - Multi Sensor Platform for Smart Building Management


Anton Köck

Materials Center Leoben, Austria





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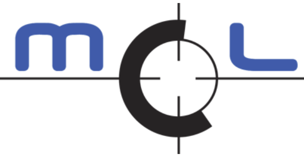


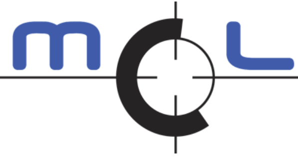
# Outline

MSP-Project

1. Motivation
2. Concept & Objectives
3. Consortium & Workpackages

Gas Sensors

4. Single Nanowire Sensors
5. Multi Nanowire Sensors
6. CMOS Integration



# 1. Motivation

## Key facts are:

- Indoor air pollution is estimated to cause ~ 2 million deaths mostly in developing countries and is supposed to pose a risk to the health of over half of the world's population,
- CO is a potential deadly indoor threat in case of defect gas heating systems and counts for a death toll of ~ 500 persons per year only in the US,
- Urban outdoor air pollution is estimated to cause ~ 1.3 million deaths worldwide per year.

There is a demand for devices enabling indoor and outdoor environmental monitoring!

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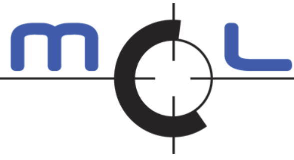

# Target Parameters

| Indoors | Outdoors |
| --- | --- |
| $CO$, $CO_2$, VOCs, PM | $NO_2$, $O_3$, $CO$, $PM_{10}$, $PM_{2.5}$, UFPs |

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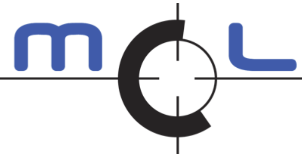
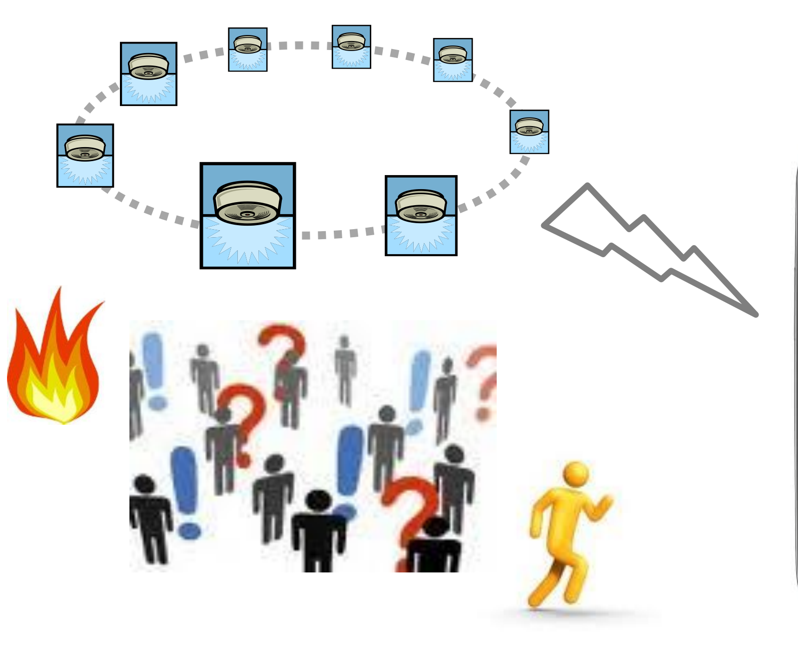


# Ideas for Applications



Sensor nodes in buildings

Wearable devices for personal environmental monitoring

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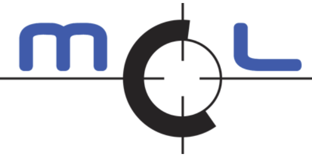
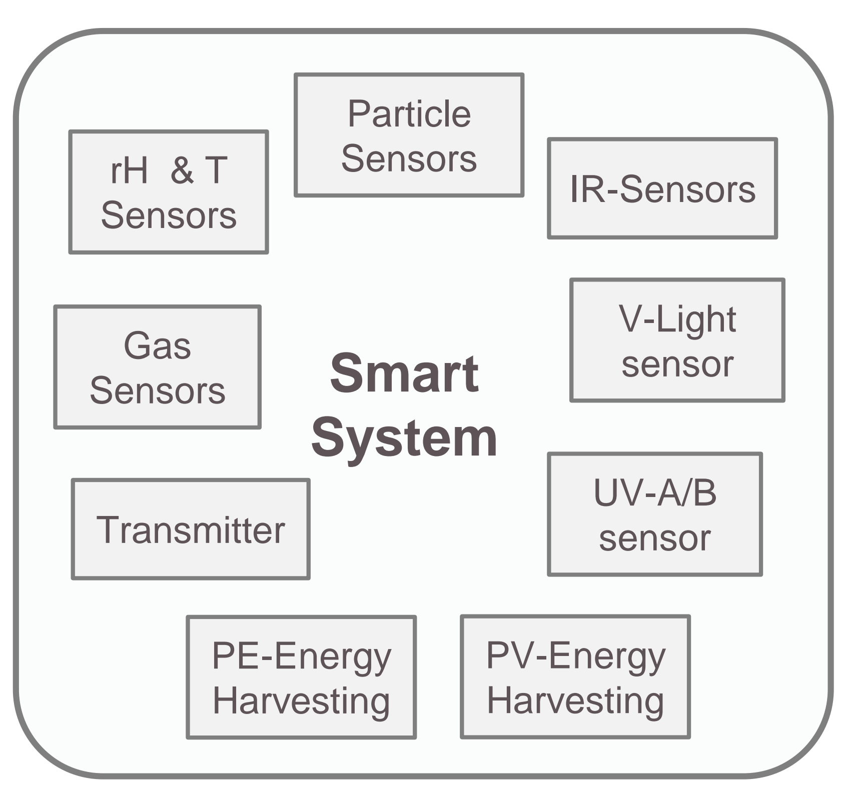


# Wish list for smart systems:

- Multi-parameter sensing
- Sensing of other parameters
- Small footprint
- Low power consumption
- Energy autonomous system
- Energy harvesting
- Wireless communication
- Cost efficient mass production
- Etc…..



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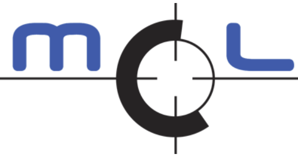
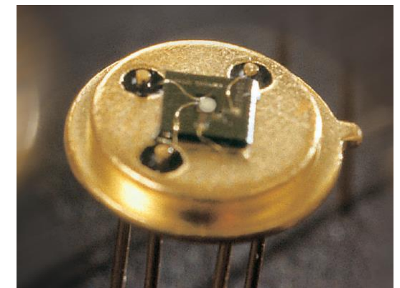


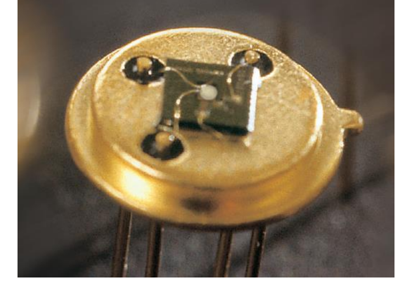
# Market situation for gas sensors

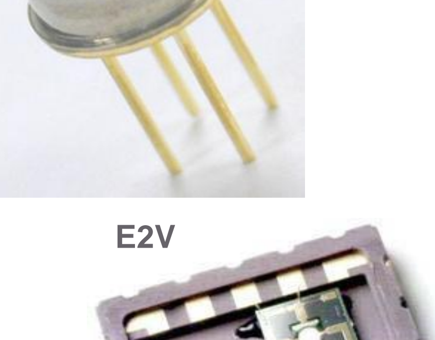
- Conventional devices
- Cross selectivities
- High power consumption
- For professional use only

Figaro Engineering

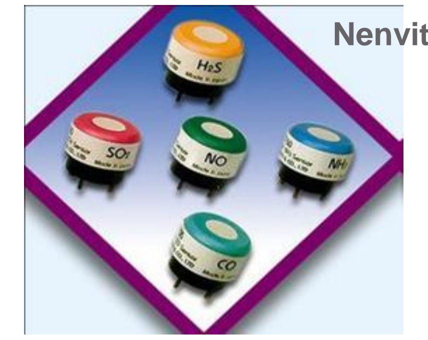
Applied Sensor



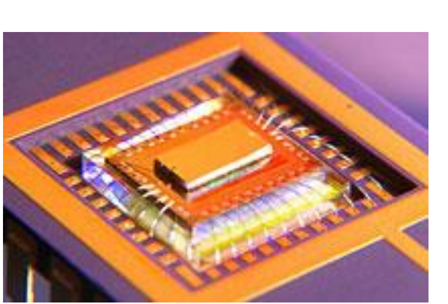




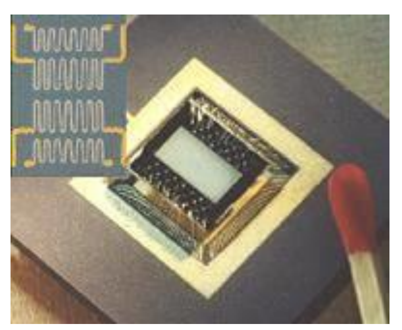
E2V

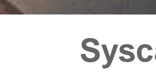


Nenvitech



Micronas





Sysca

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GA no: 611887

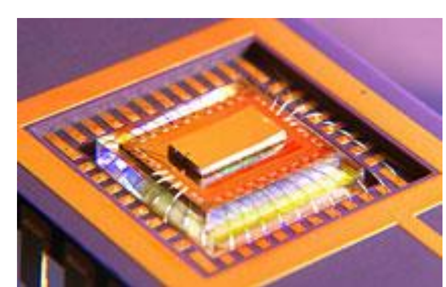
# Market situation for gas sensors

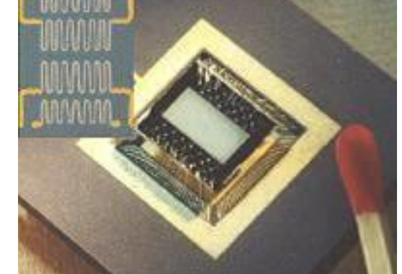
- Conventional devices
- Cross selectivities
- High power consumption
- For professional use only

**Figaro Engi**

Today‘s gas sensors are not smart devices – no integration with CMOS technology!

**Nenvitech**





**Micronas**

**Sysca**

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# Development of smart MSP systems providing personal environmental monitoring indoors & outdoors is a huge challenge!

MSP
MSP
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MSP
MSP

12:03
Air Quality
12.12.2012
12:12
Tech Gate
Room 2.12
$CO_2$
VOC
$CO_2$ 4
CO 2
$NO_2$ 1
VOC 4
$O_3$ 2

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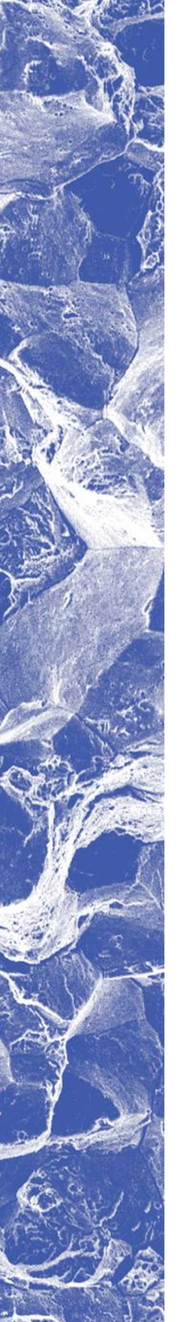

# High commercial interest in smart gas sensor devices for HVAC control: CO, $CO_2$, VOCs

**40%** of European energy consumption used in buildings

**50%** of energy require-ments relate to heating / cooling

**60%** of Europe's building stock is over 25 years old

**Air quality regulated HVAC might save ½ of energy !**
**Potential to save ~ 25% of buildings energy need !**

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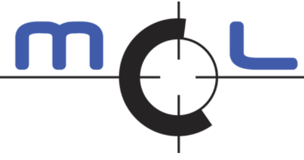
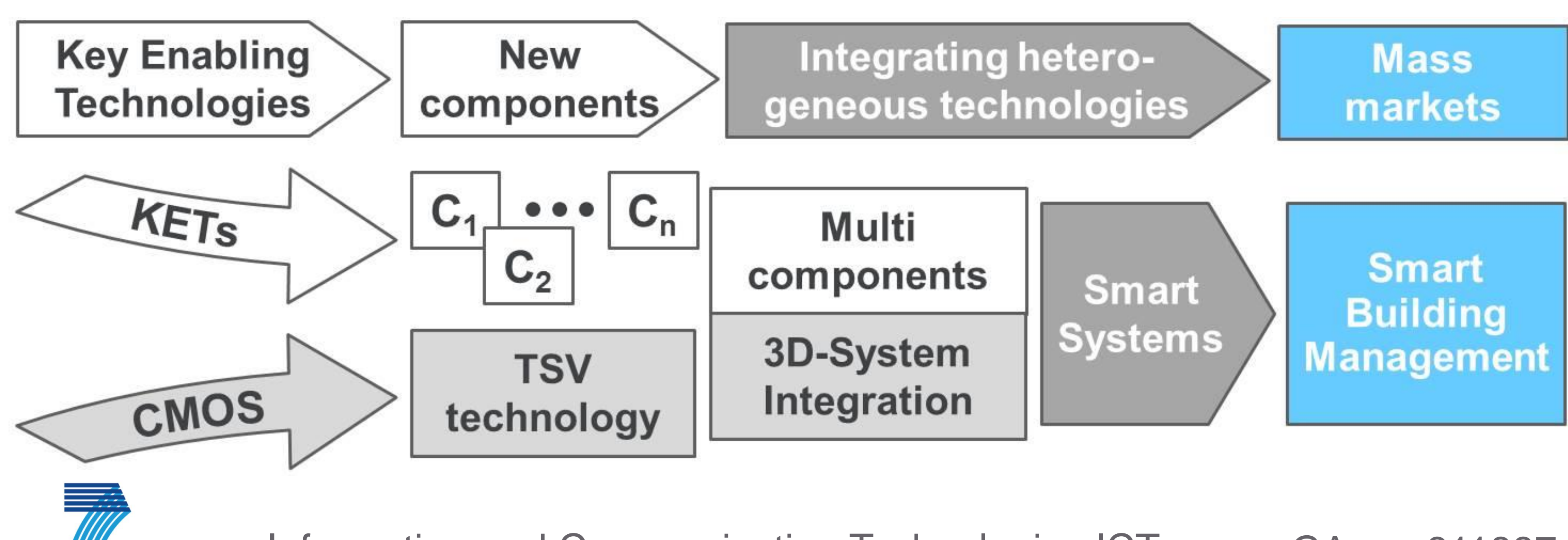
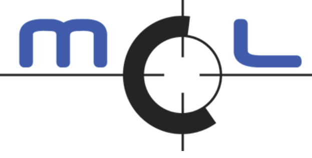


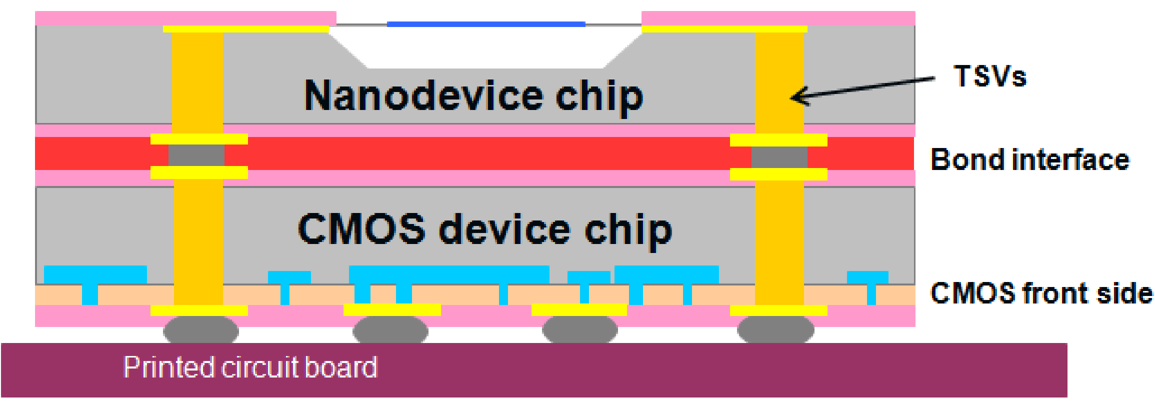
# 2. Concept & Objectives

- Take-up of Key Enabling Technologies (Nano,...) for new components and devices
- CMOS technology as sound foundation to ensure cost efficient mass fabrication
- Integrating heterogeneous technologies for realization of smart systems





- Development of process & manufacturing chains enabling take-up of KETs and multi-project-wafer approach
- Development of novel components & devices for 3D-integration
- Development of wireless communication for networks and handheld devices
- Realization of specific 3D-integrated MSP demonstrator systems



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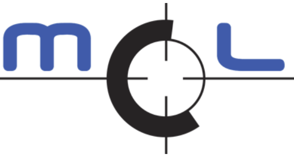


# 3. Consortium & Workpackages

- 17 partners from 6 countries

UNIVERSITY OF OXFORD

UNIVERSITY OF CAMBRIDGE

CAMBRIDGE CMOS SENSORS

THE UNIVERSITY OF WARWICK

SAMSUNG

Holst Centre

boschman

Fraunhofer

SIEMENS

AppliedSensor

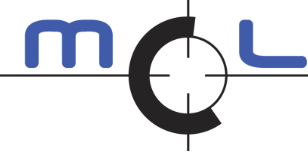
FMF

amu

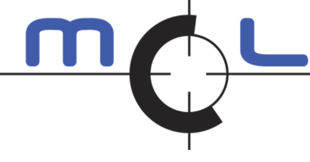
EVG

UCL Université catholique de Louvain

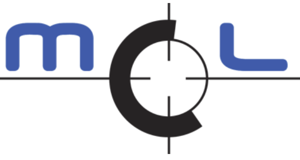
vito vision on technology



| 1 | Materials Center Leoben Forschungs GmbH | Austria |
|---|---|---|
| 2 | ams AG | Austria |
| 3 | AppliedSensor GmbH | Germany |
| 4 | Boschman Technologies B.V. | Netherlands |
| 5 | University of Oxford | UK |
| 6 | Cambridge CMOS Sensors | UK |
| 7 | E V GROUP E. THALLNER GMBH | Austria |
| 8 | Fraunhofer-Gesellschaft zur Foerderung der Angewandten Forschung e.v | Germany |
| 9 | Stichting imec Nederland (Holst Centre) | Netherlands |
| 10 | ALBERT-LUDWIGS-UNIVERSITAET FREIBURG: The Freiburg Materials Research Center (FMF) | Germany |
| 11 | Siemens AG | Germany |
| 12 | University of Cambridge | UK |
| 13 | University Louvain | Belgium |
| 14 | Universitá degli studi di Brescia | Italy |
| 15 | University of Warwick | UK |
| 16 | VITO - VLAAMSE INSTELLING VOOR TECHNOLOGISCH ONDERZOEK N.V. | Belgium |
| 17 | Samsung Electronics UK Ltd. | UK |



| | |
|---|---|
| Siemens AG | Large company |
| Samsung Electronics UK Ltd. | Large company |
| ams AG | Large company |
| E V GROUP E. THALLNER GMBH | Large company |
| AppliedSensor GmbH | SME |
| Boschman Technologies B.V. | SME |
| Cambridge CMOS Sensors | SME |
| Fraunhofer-Gesellschaft zur Foerderung der Angewandten Forschung e.v | R&D institute |
| Materials Center Leoben Forschung GmbH | R&D institute |
| Stichting imec Nederland (Holst Centre) | R&D institute |
| VITO - VLAAMSE INSTELLING VOOR TECHNOLOGISCH ONDERZOEK N.V. | R&D institute |
| ALBERT-LUDWIGS-UNIVERSITAET FREIBURG: The Freiburg Materials Research Center (FMF) | University |
| Universitá degli studi di Brescia | University |
| University of Cambridge | University |
| University Louvain | University |
| University of Oxford | University |
| University of Warwick | University |



# Value Chain

KETs

CMOS

$C_1$ ••• $C_n$

$C_2$

TSV technology

Multi components

3D-System Integration

Smart Systems

Smart Building Management

THE UNIVERSITY OF WARWICK

UCL Université catholique de Louvain

UNIVERSITY OF OXFORD

UNIVERSITY OF CAMBRIDGE

Holst Centre

AppliedSensor

CAMBRIDGE CMOS SENSORS

SIEMENS

vito vision on technology

SAMSUNG

Fraunhofer

ams

EVG

boschman

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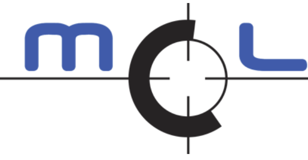
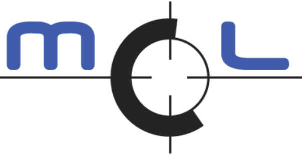


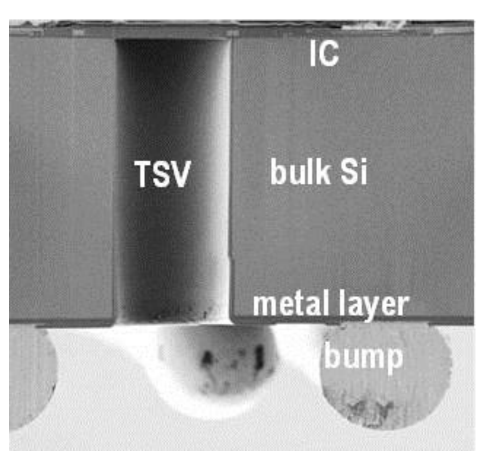
# Work Package Overview

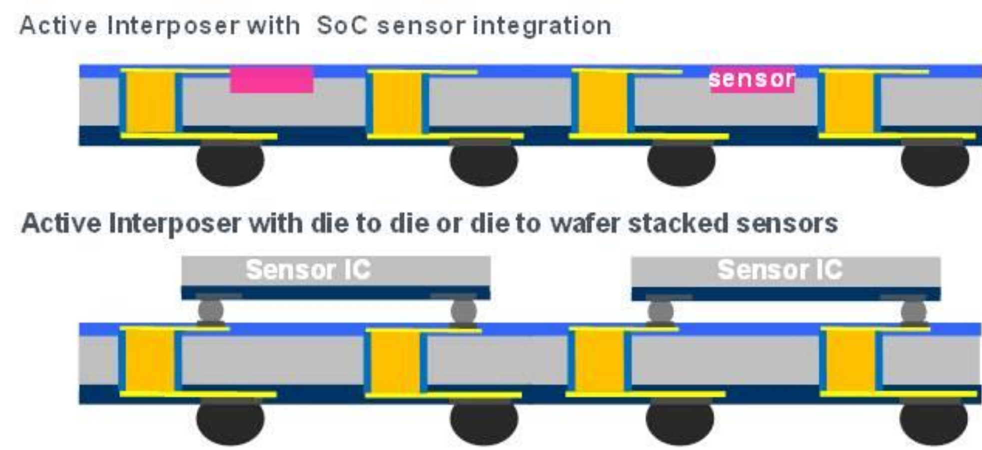
| WP | Work Package Description | Participant organisation name | Work Package Leader |
|---|---|---|---|
| 1 | Management | MCL | Anton Köck |
| 2 | Overall Concept Development | ams AG | Martin Schrems |
| 3 | Development of Components and Devices | AppliedSensor GmbH | Heiko Ulmer |
| 4 | Characterization and Test of Components and Devices | Siemens AG | Oliver von Sicard |
| 5 | Development of CMOS Platform Chip | Fraunhofer IISB & IIS | Markus Stahl-Offergeld |
| 6 | Data processing and wireless communication | Holst Centre IMEC the Netherlands | Guido Dolmans |
| 7 | Fabrication of 3D-integrated Demonstrator Systems | ams AG | Martin Schrems |
| 8 | Performance Evaluation of Demonstrator Systems | Vito NV | Jan Theunis |
| 9 | Exploitation | ams AG | Karin Ronijak |
| 10 | Dissemination | University of Cambridge | Florin Udrea |

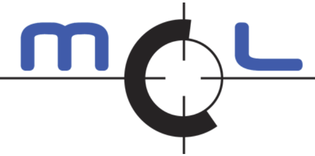


# WP2: Overall Concept Development

- Overall concept development of process and manufacturing chains for all components and devices, which enable the take up of key enabling technologies and are capable for a multi-project wafer approach
- All developments based on TSV technology to ensure the capability for 3D-integration

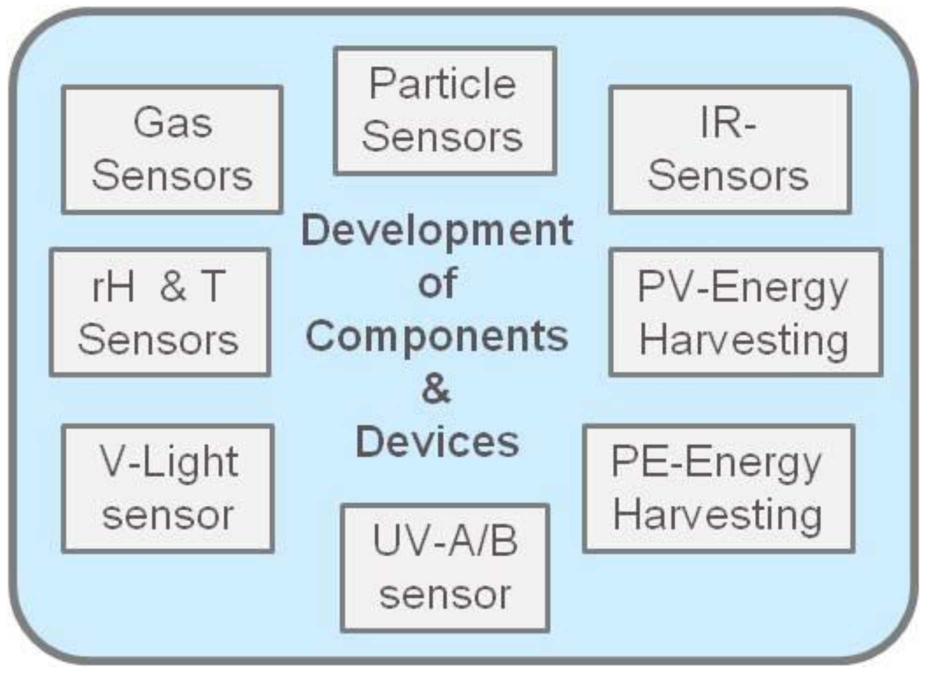




# WP3: Development of Components & Devices

- The specific multi-project wafer approach shall enable the development of entirely new components and devices based on KETs, which require a cost efficient and flexible industrial approach on a wafer scale in order to reach the target markets

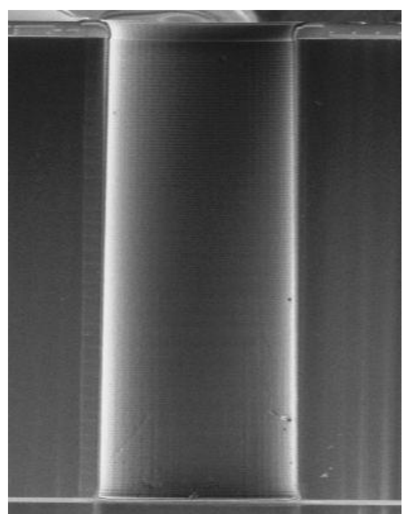


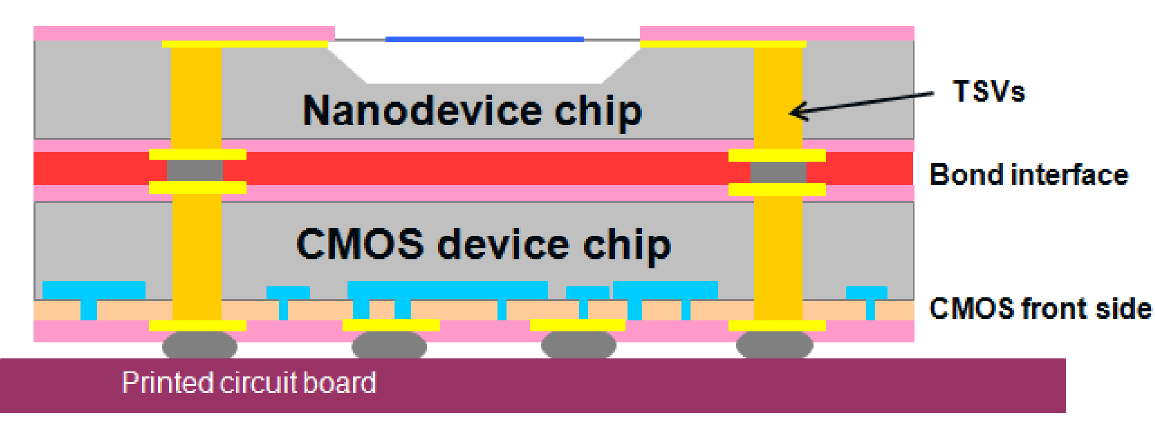
# WP4: Characterization & Test of Components & Devices

- Characterization and test of components and devices in laboratory and in-field: gas measurements, optical, electrical, mechanical characterization, energy harvesting,…etc.

# WP5: Development of CMOS Platform Chip

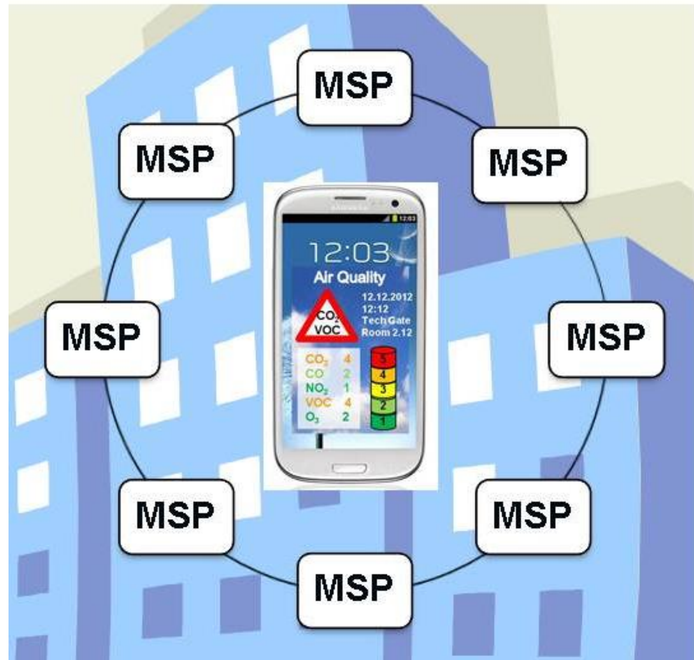
- Development of a robust and ultra-low-power CMOS platform chip with standardized TSV design which enables the flexible “plug-and-play” 3D-integration of the components to customer specific sensor systems wherever feasible and commercially justified.





# WP6: Data Processing & Wireless Communication

- Development of data processing and wireless communication to enable ultra-low power consumption compatible with energy harvesting operation. The wireless communication part is key to achieve a low-power, robust and stable link between mobile and fixed nodes.



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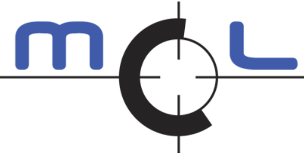
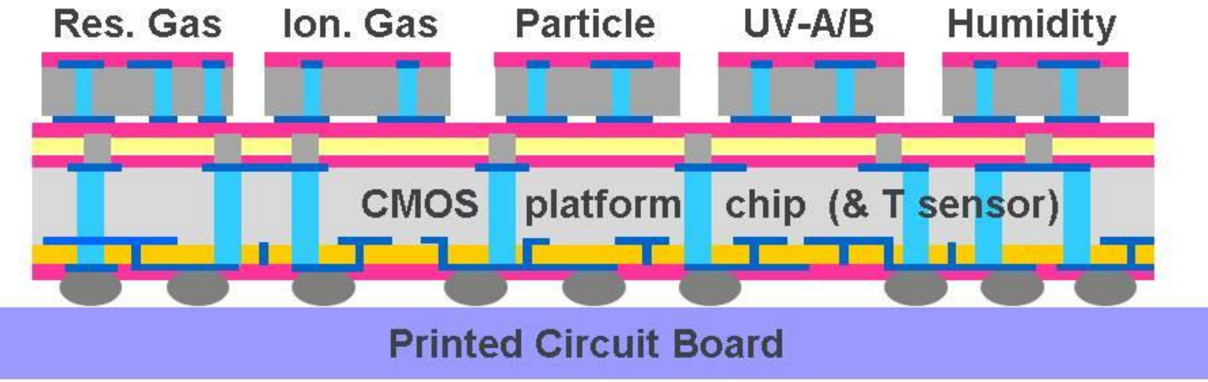


# WP7: Fabrication of 3D-integrated Demonstrator Systems

- Development of 3D-integrated demonstrators, processes and technologies for stacking all components and devices. The key building block is the TSV & BRDL structure (active/passive Si interposer) that can be used as the basic "LEGO™" building block for 3D-integration to MSP demonstrator systems for:
- Smart Building Management
- Outdoor environmental monitoring



# WP8: Performance Evaluation of Demonstrator Systems

- Testing the performance of all MSP demonstrator devices. This is a critical issue, because all sensors have to be tested in all specific test environments, for example a UV sensor based on NWs has to be insensitive against all target gases in the gas test rig.

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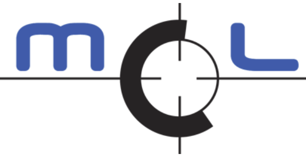
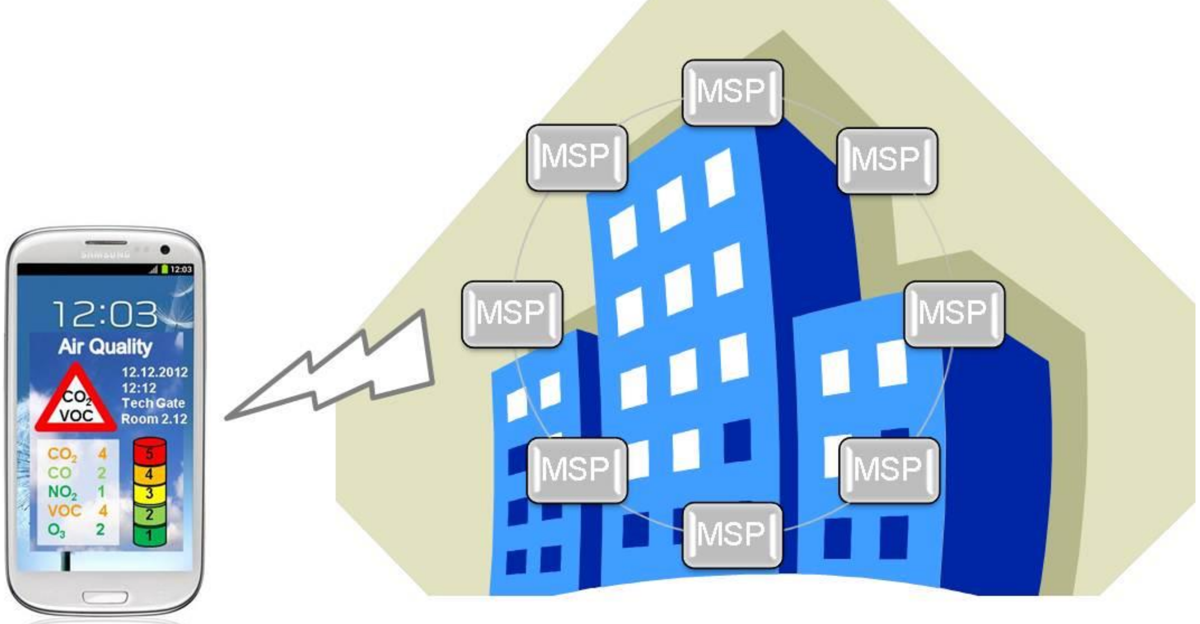
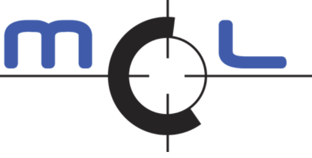


# WP9: Exploitation

- In order to increase the impact of the MSP project, exploitation is planned on several levels ranging from finding new industrial partners for application to and exploiting the 3D-stacking technologies to offering an MPW service for sensing functions, similar to the current one for ASICs.





# WP10: Dissemination

- The objective of this work package is to disseminate the project results by all appropriate academic, industrial and commercial channels and to scientific publications policy at selected conferences and in journals with high impact factors. Target is a minimum of 20 publications in conferences and journals.
- Most important goal is to strengthen the International and European visibility of the MSP project and establishing links for exchanges and collaborations with other subject-related EU projects.
- We have already organized the international NANOSENS conference (2007, 2009, 2010, www.nanosens.at), where focus is on industrial applications of nanosensors and their potential for 3D-sensor integration. In order to utilize synergies and save resources the established NANOSENS conference platform will be used for dissemination of the MSP project results.

# Metal Oxide Nanowire Gas Sensors for Indoor and Outdoor Environmental Monitoring

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F. Schrank[d], M. Schrems[d], J. Siegert[d], J. Teva[d]
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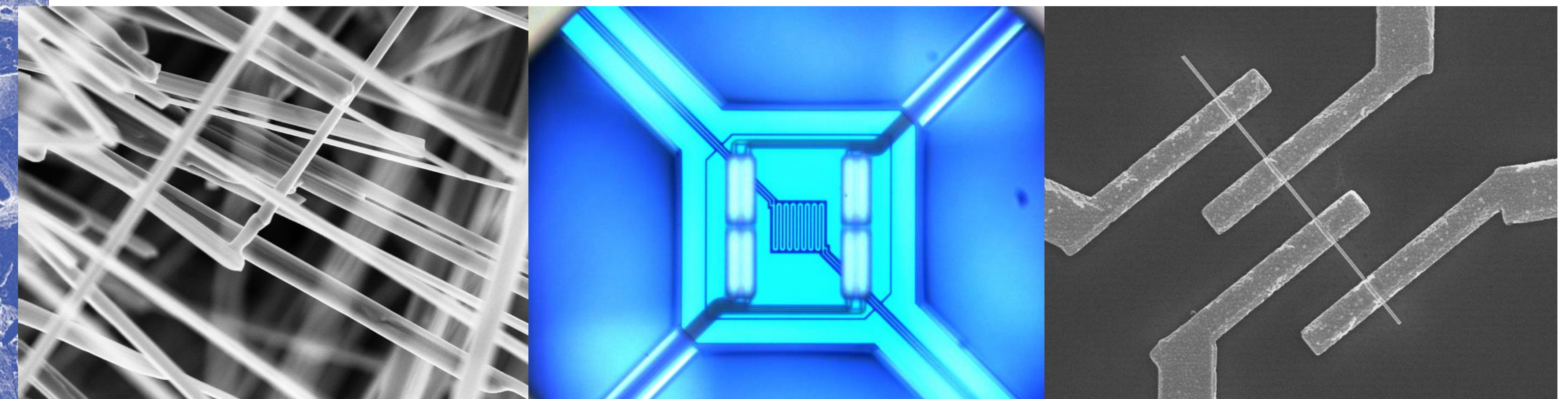
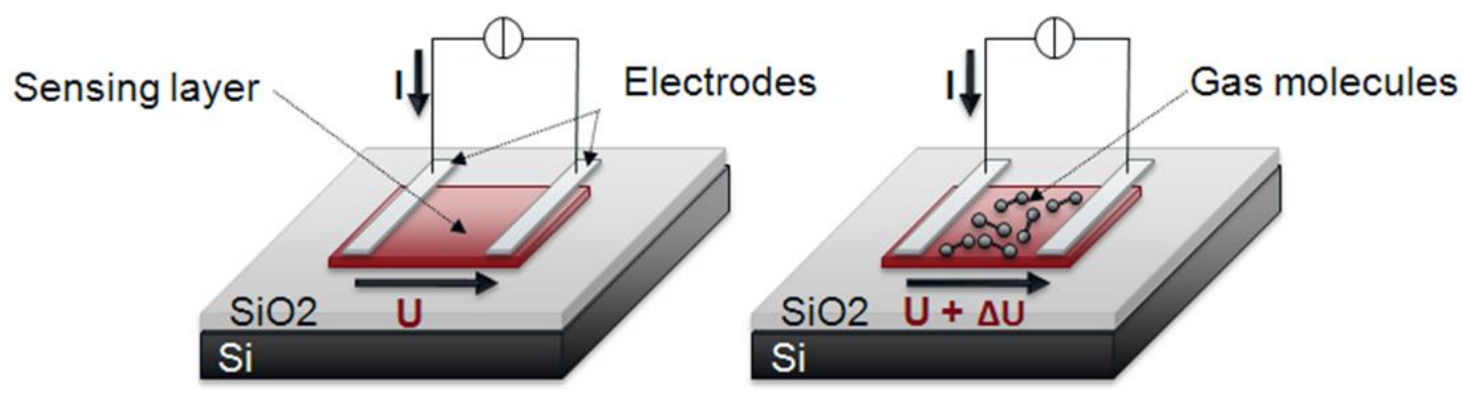
[c]Siemens AG, Munich, Germany
[d]ams AG, Unterpremstaetten, Austria

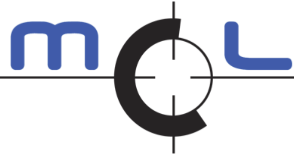


# Thin film and nanowire sensors

- $SnO_2$-thin films
- $SnO_2$-NWs (n-type)
- CuO-NWs (p-type)
- ZnO-NWs (n-type)

Target gases:
CO, $H_2$, $H_2S$,
$CO_2$, VOCs, $NO_2$
In dry and humid air





# 4. Single Nanowire Sensors

- $SnO_2$-NWs

1st step: spray pyrolysis

Liquid pressure

Air atomizing nozzle

$SnO_2$ layer formed by:
$SnCl_4 + H_2O \rightarrow SnO_2 + HCl$

On/Off pressure

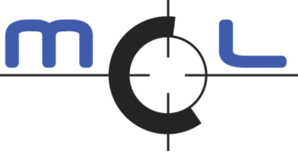
Substrate:
Si + 40nm CuO

Air pressure

500° C

Solution of $SnCl_4 \cdot 5H_2O$

Hot plate

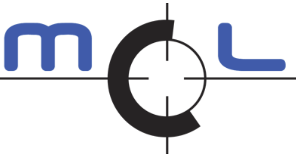


## $2^{nd}$ step: annealing process 900°C

- 900°C in Ar at atmospheric pressure - no vacuum required !
- „Conversion“ of nanocrystalline film to single crystalline NWs

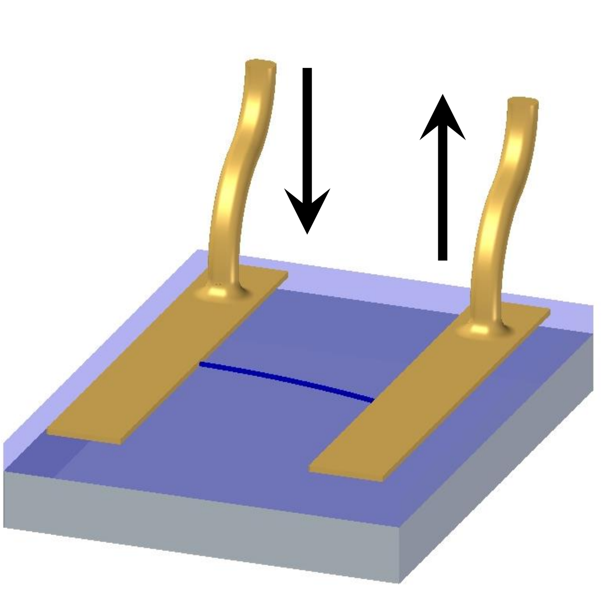
10 µm
EHT = 5.00 kV
WD = 10.4 mm
Mag = 1.00 K X
Stage at T = 70.0 °
Signal A = InLens
Time :11:19:31

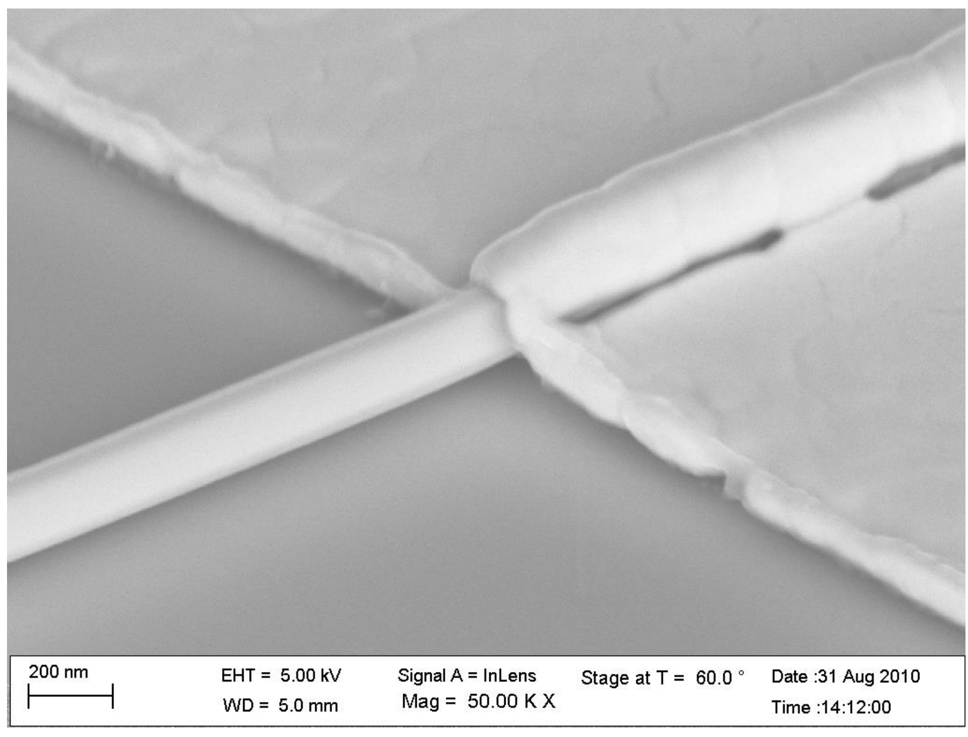
5 nm

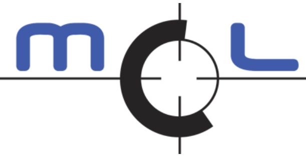


## 3rd step: Transfer of $SnO_2$-nanowires to $SiO_2$/Si-Substrate

- Spin-coating on Si-chips
- Photolithography or / and e-beam lithography
- 200 nm Ti-Au + lift-off for contacts to NWs





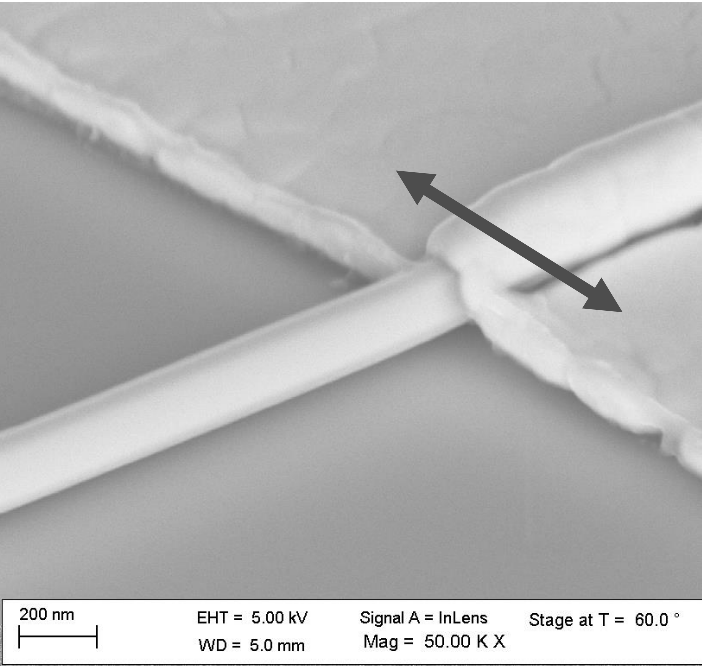


# Single NW-devices

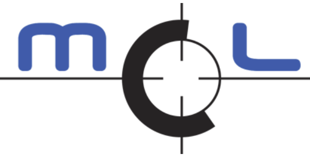
- $SnO_2$ NW sensor fabricated by optical lithography
- 2-point measurement
- L ~ 53 µm, diameter ~ 300 nm

- $SnO_2$ NW sensor fabricated by e-beam lithography
- 4-point measurement
- L ~ 345 nm, diameter ~ 80 nm

# Reliability issues!!

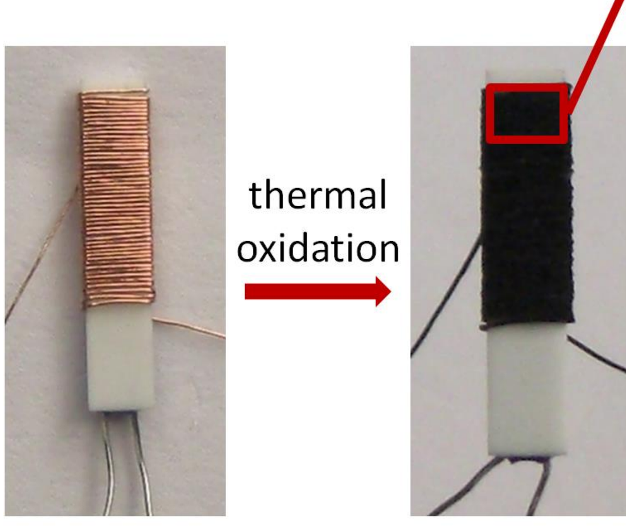


**Centre for Electron Microscopy Graz**

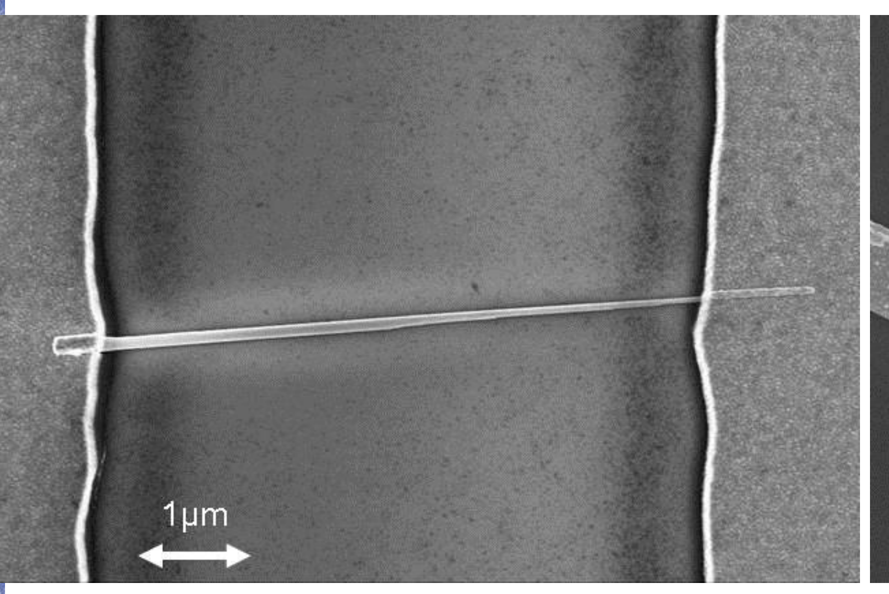


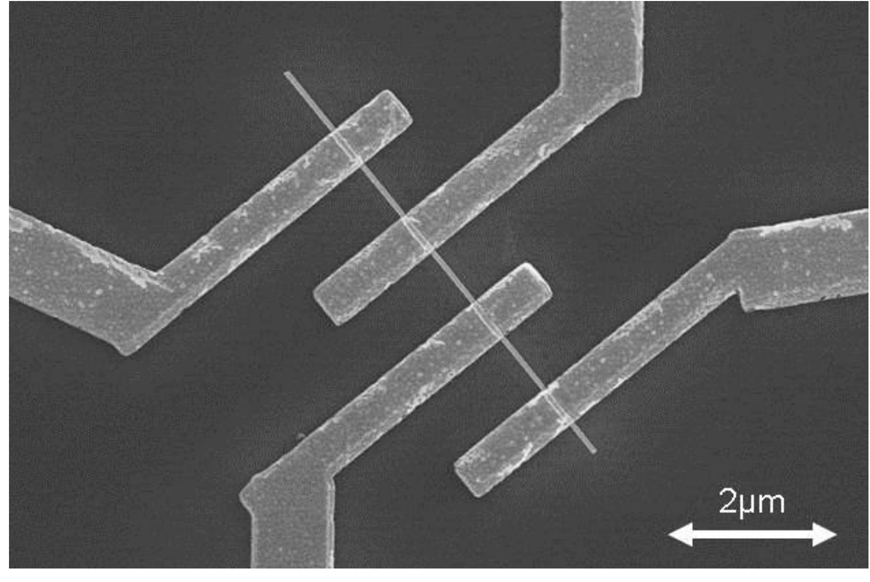
## CuO-NWs (& ZnO-NWs)

- Thermal oxidation of Cu-wires
- Resistive heating on Pt100
- NW sample in Isopropanol
- Spin-coating on Si-chips



# Single NW-devices

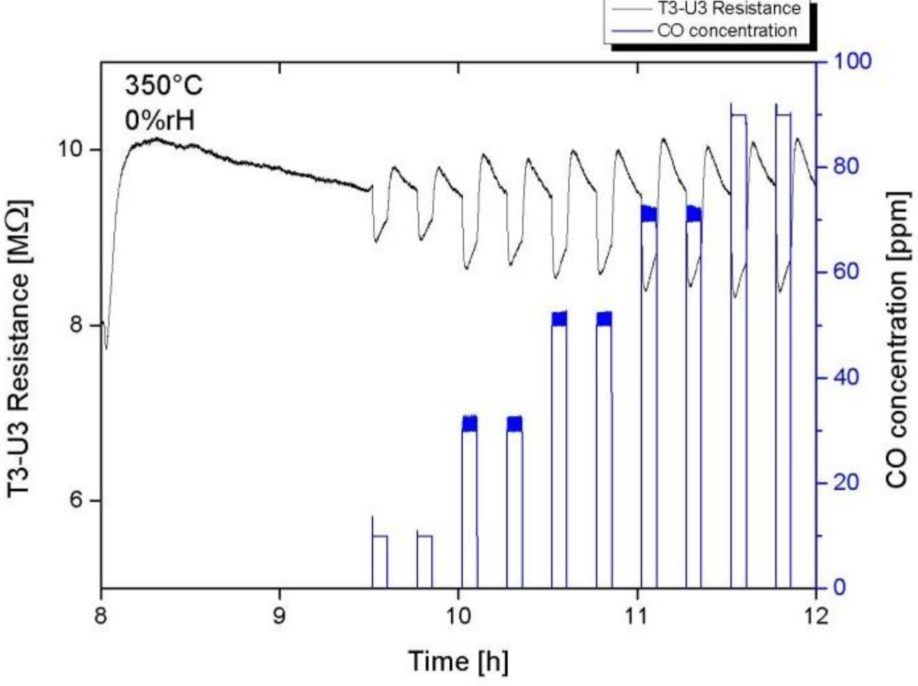



- CuO NW sensor fabricated by optical lithography
- 2-point measurement
- L ~ 6,5 µm, diameter ~120 nm

- CuO NW sensor fabricated by e-beam lithography
- 4-point measurement
- L ~ 900 nm, diameter ~ 70 nm

- Response of $SnO_2$-NW sensor to CO
- T = 350°C




| CO concentration | Response 350°C dry synthetic air [%] | Response 350°C humid synthetic air [%] |
|---|---|---|
| 10 ppm | 6,0 | 2,4 |
| 30 ppm | 8,7 | 3,5 |
| 50 ppm | 9,3 | 4,8 |
| 70 ppm | 11,5 | 5,6 |
| 90 ppm | 12,4 | 6,1 |

- Response of CuO-NW sensor to CO
- T = 350°C

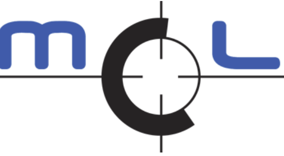
dry
50% r.H.

nanowire resistance [MΩ]
3.50
3.25
3.00
2.75
2.50
2.25
2.00
1.75
1.50

CO concentration [ppm]
200
175
150
125
100
75
50
25
0

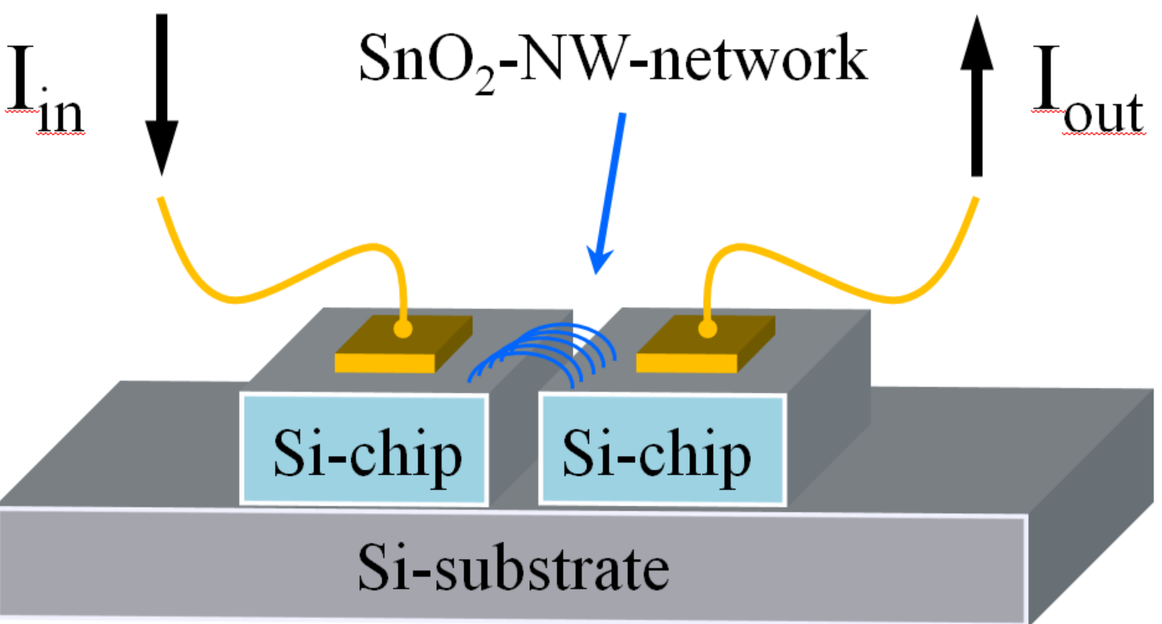
0
50
100
150
200
time [min]

—nanowire resistance
—CO concentration



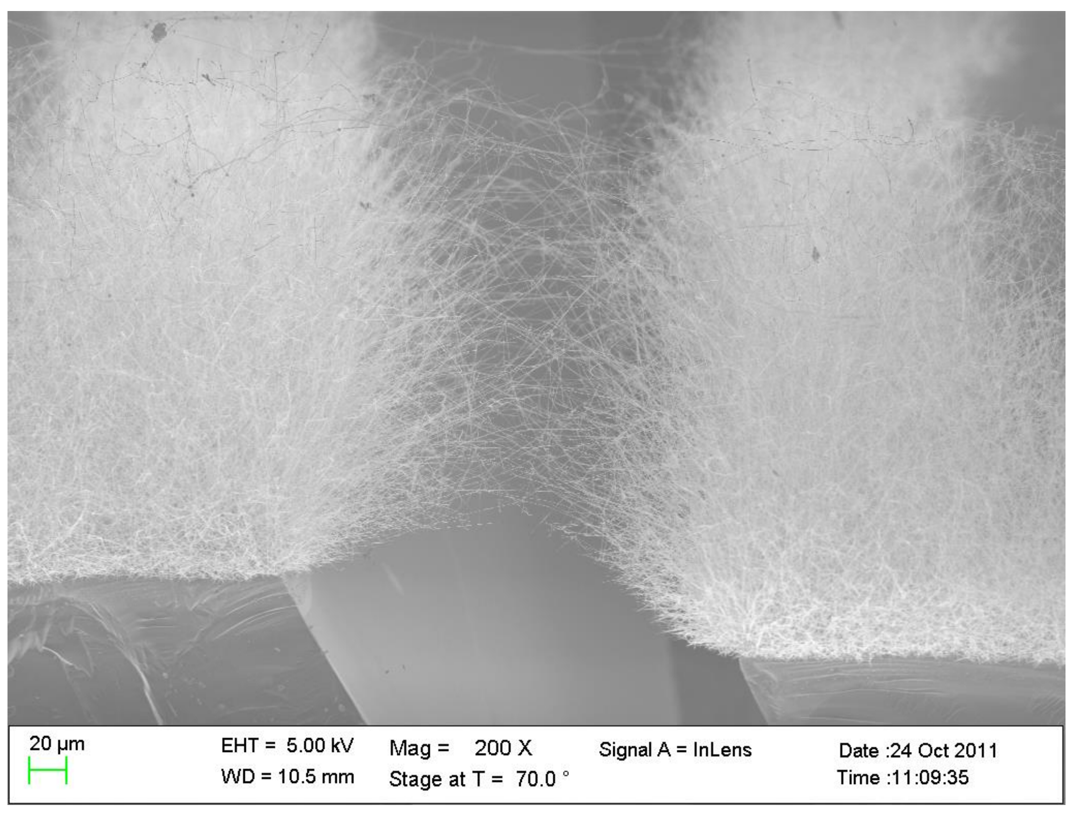
# 5. Multi Nanowire Sensors

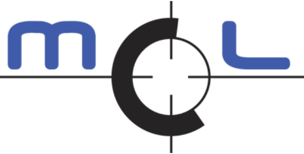
- $SnO_2$-NWs
- Spray pyrolysis for $SnO_2$ layer
- Annealing process



- $SnO_2$-NWs

- Spray pyrolysis for $SnO_2$ layer
- Annealing process



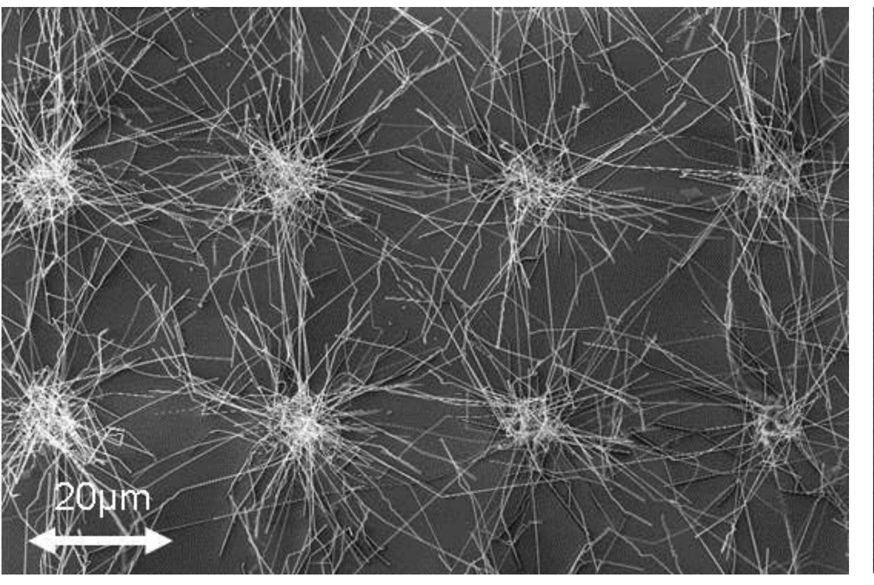


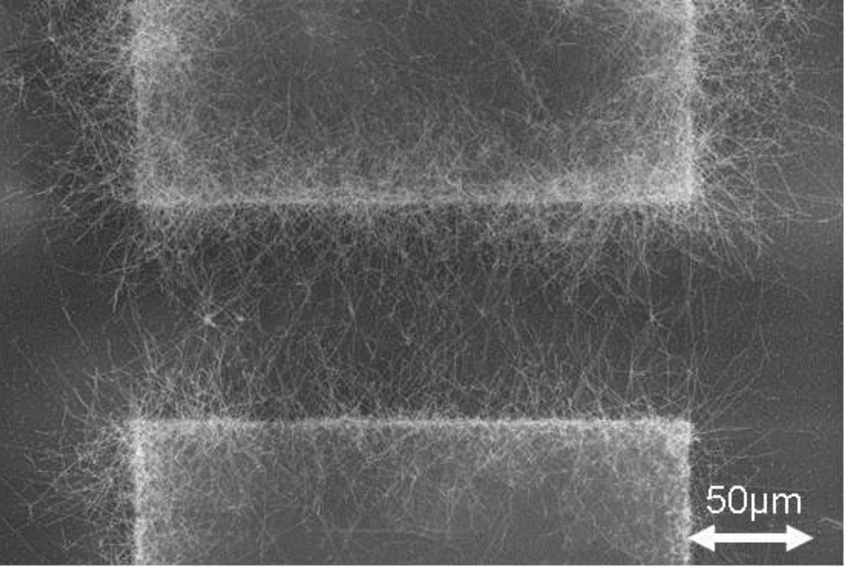
- $SnO_2$-NWs
- Response to 10 – 100 ppm $H_2$ in dry synthetic air at 300°C

10 ppm
20 ppm
30 ppm
40 ppm
50 ppm
60 ppm
70 ppm
80 ppm
90 ppm
100 ppm
Sensor Resistance [GΩ]
0.65
0.60
0.55
0.50
H2 flow [sccm]
300
250
200
150
100
50
0
0
3600
7200
10800
14400
18000
21600
25200
28800
Time [s]

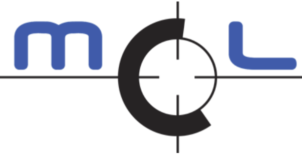
- $SnO_2$-NWs

## NW integrated on Si-chips

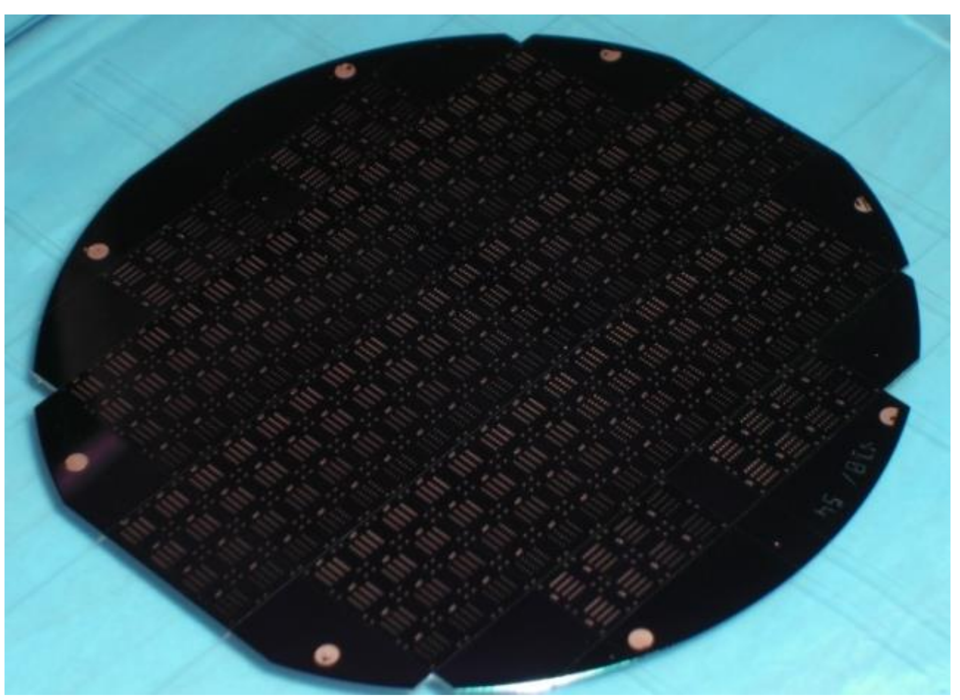


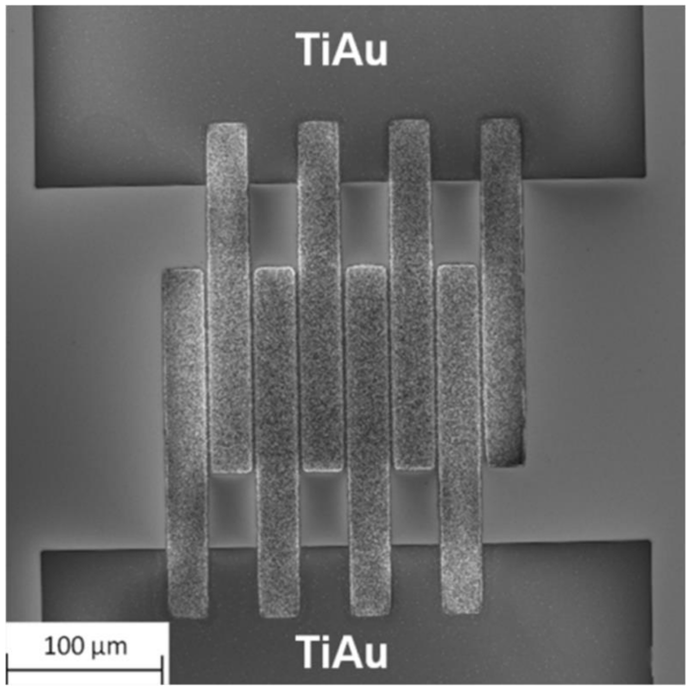



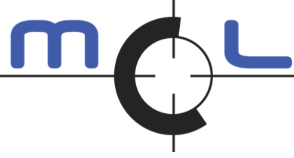

- $SnO_2$ nanowires locally synthesized on 10 x 10 µm$^2$ sized metal squares
- $SnO_2$ nanowires locally synthesized on 200 µm wide metal stripes



- CuO-NWs
- Cu-structures on 6”-Si-wafer
- Various designs with different spacing
- Oxidation in synthetic air $T < 400^{o}C$







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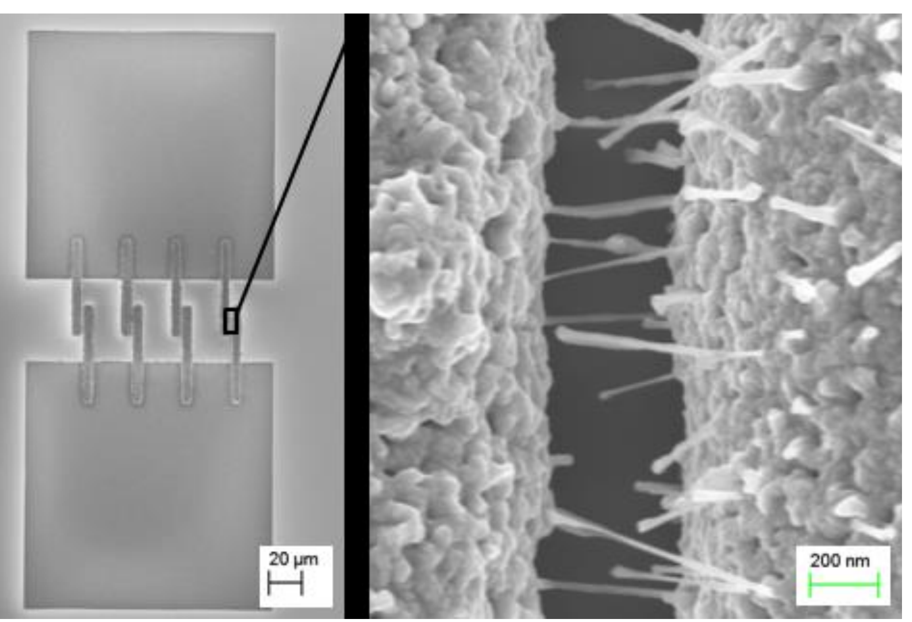
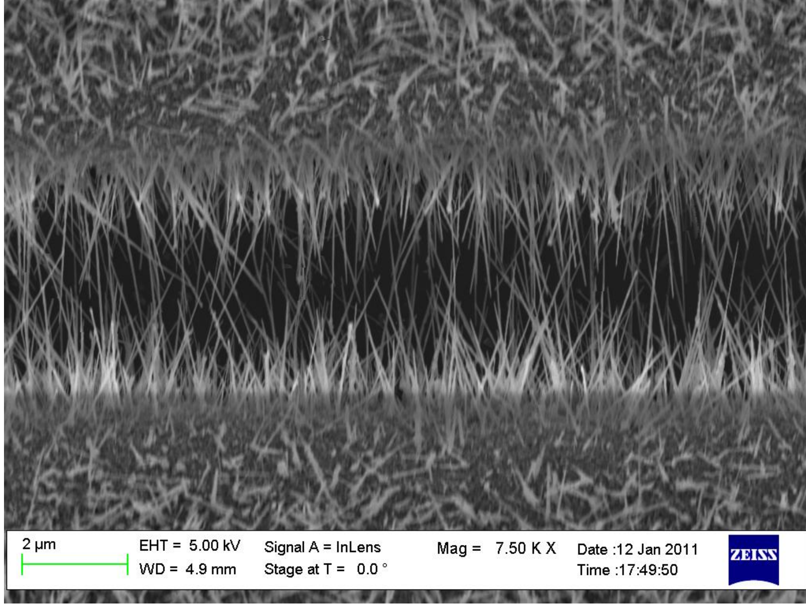


# Gas sensing result $H_2S$

sensor resistance

$H_2S$ concentration

10 ppb

sensor resistance [kΩ]

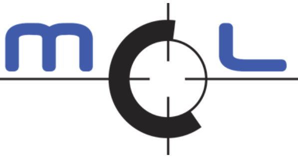
800
700
600
500
400
300
200

$H_2S$ concentration [ppb]

600
500
400
300
200
100
0

0 20 40 60 80 100 120 140 160

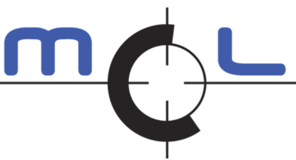
time [min]



# 6. CMOS Integration

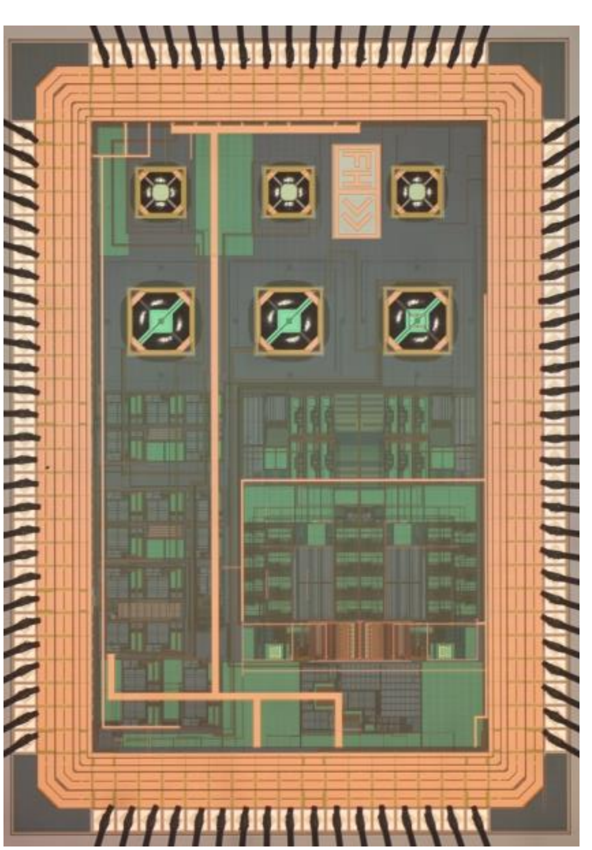
- Implementation of heating and temperature control
- CMOS fabricated micro-hotplates
- Poly-Si heater & and thermistor

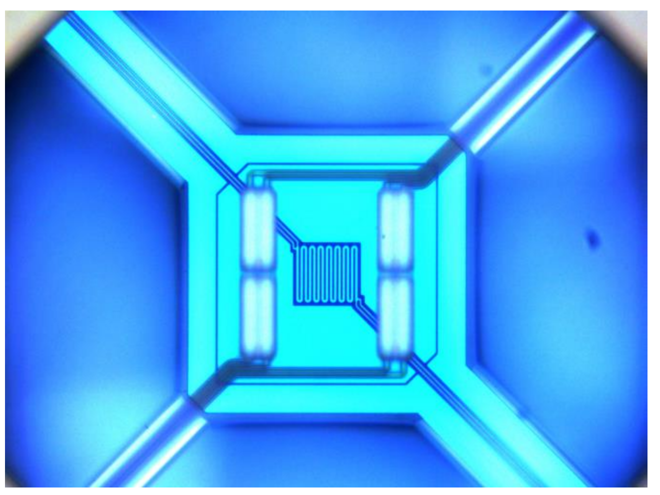
20 µm
EHT = 1.00 kV
WD = 6.9 mm
Signal A = SE2
Mag = 387 X
Date :30 Nov 2012
Time :10:26:38
ZEISS

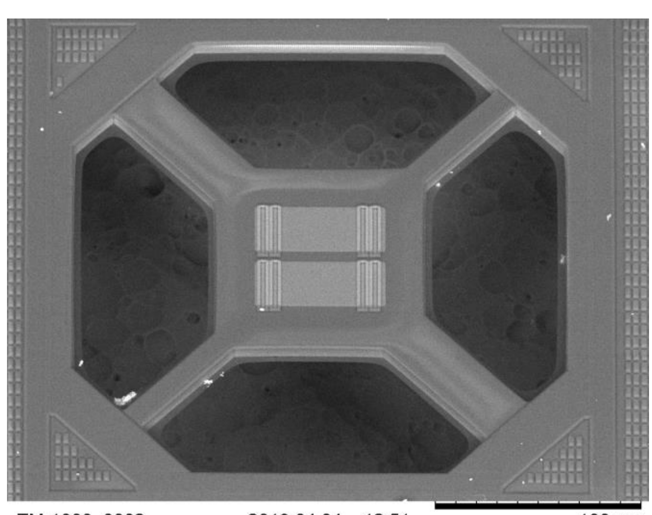


# $SnO_2$ thin film sensors

- Implementation on µ-hps
- Fully CMOS integrated

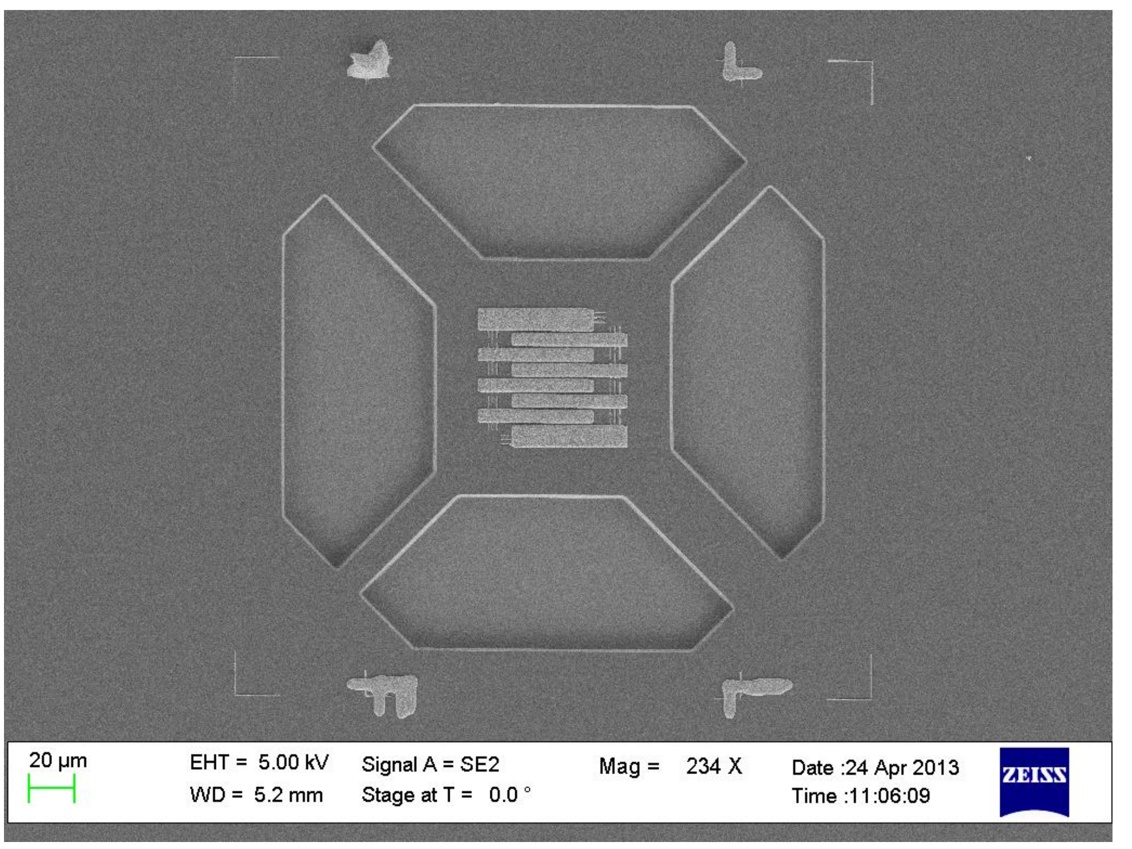


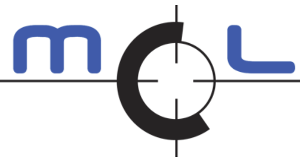




# Integration of NWs on µ-hot plate

- $SnO_2$-NWs require transfer process !
- But: CuO and ZnO-NWs can be synthesized on µ-hps !





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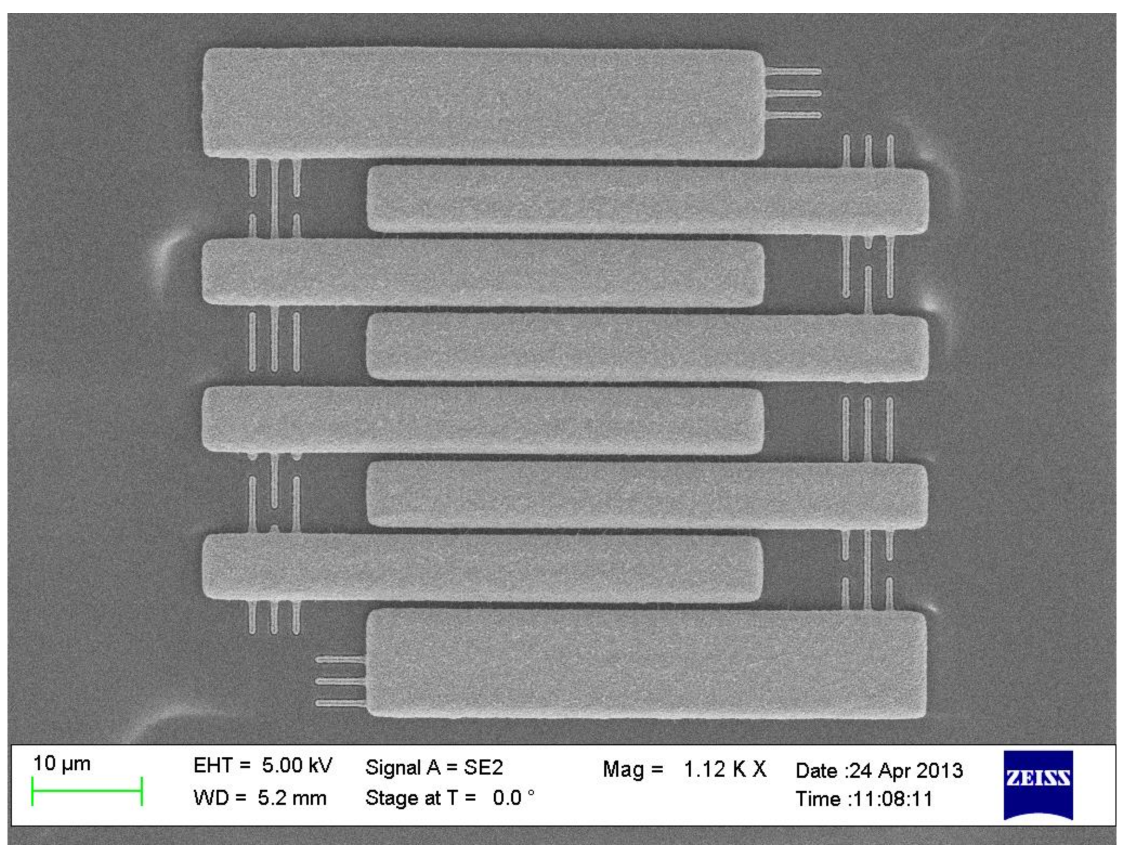

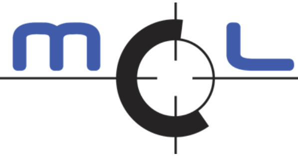

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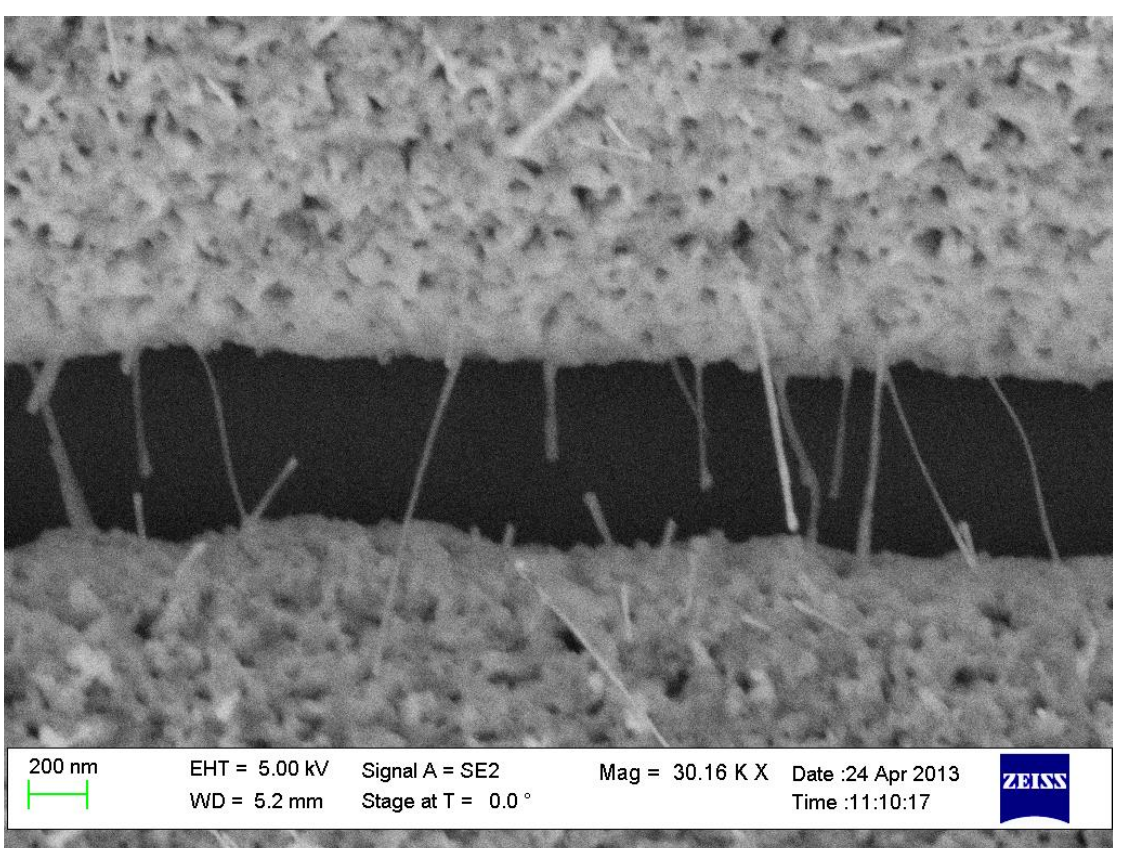
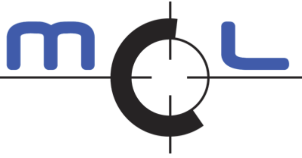
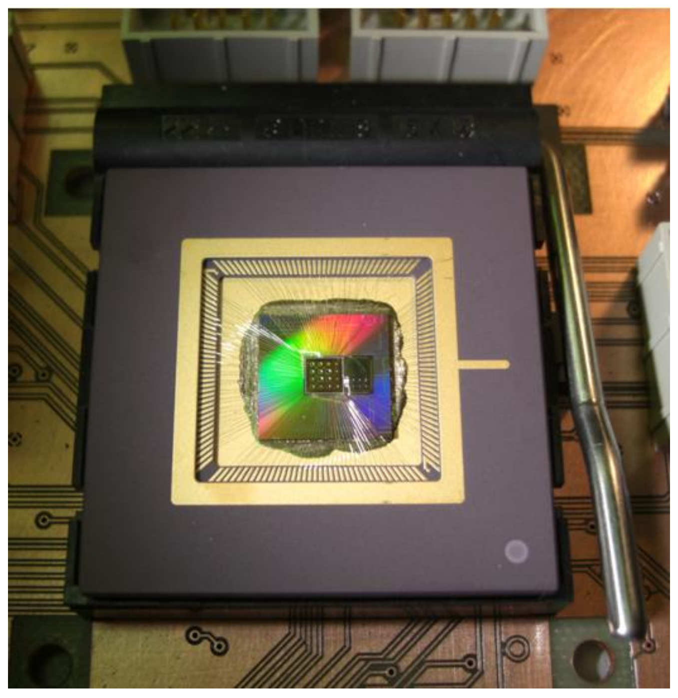
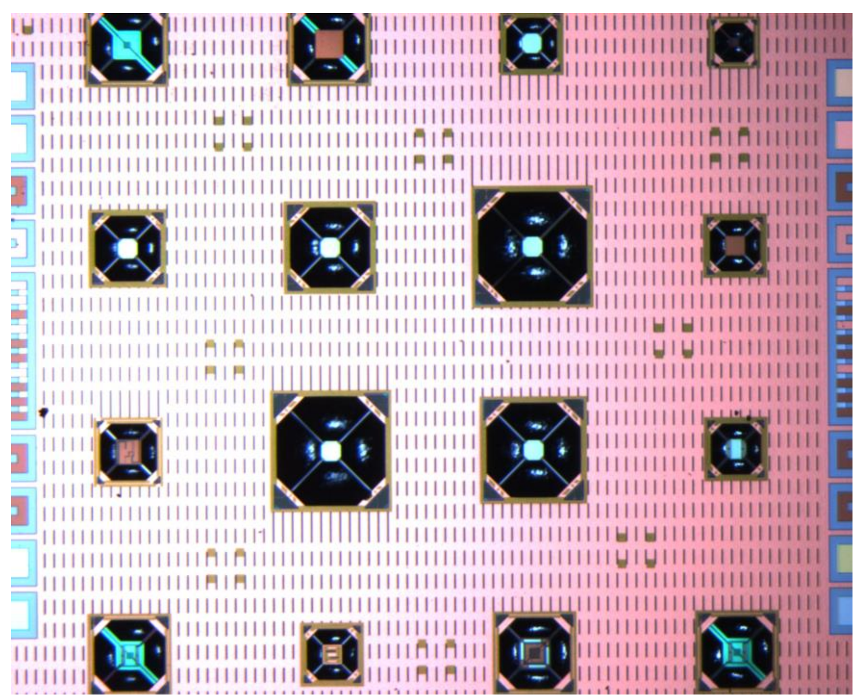


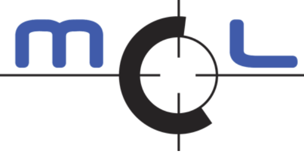


# Vision: Multi-parameter gas sensor array

- 16 µHPs on a single chip
- Mix of $SnO_2$ thin film, $SnO_2$, CuO, and ZnO-NW sensors
- Functionalization with NPs for improved selectivity

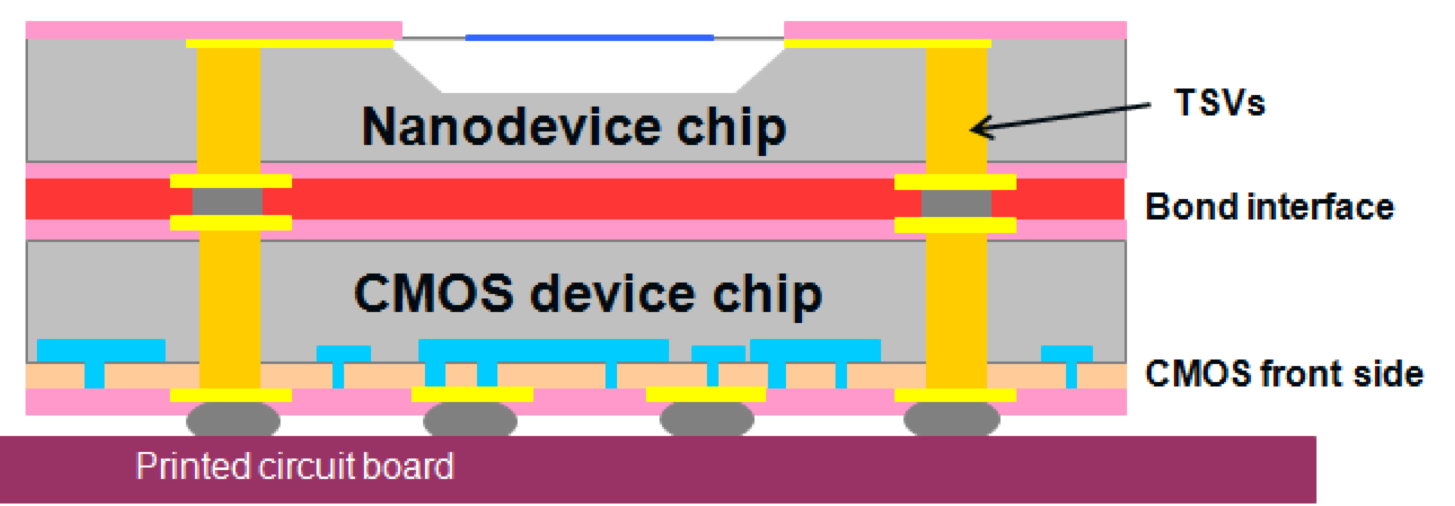


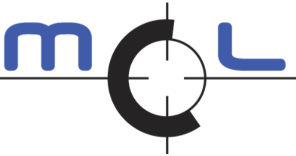




# 3D-SiP-Integration

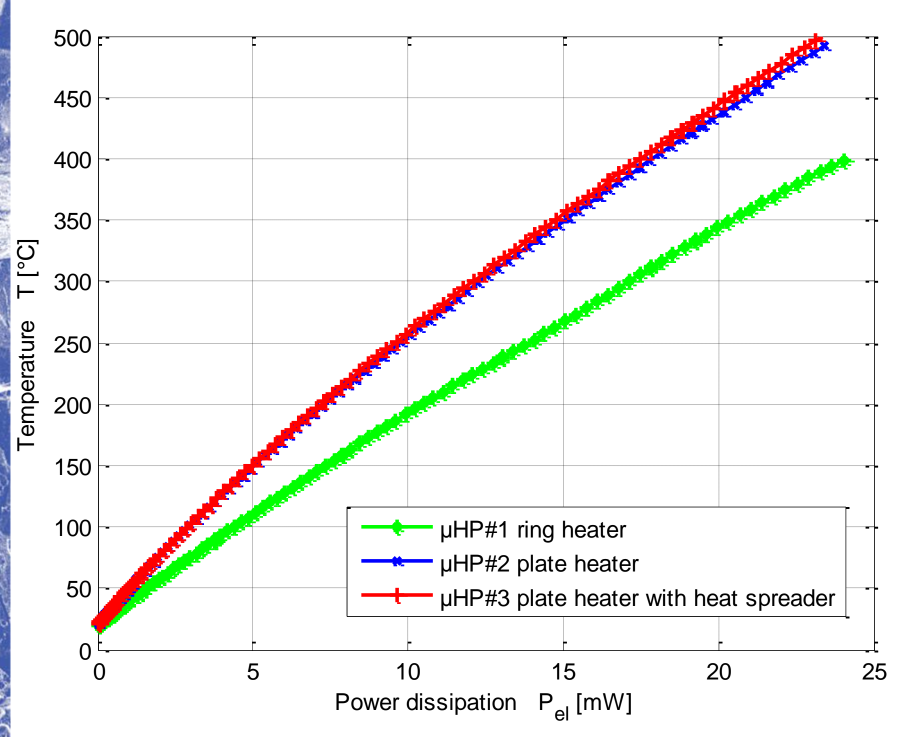
- COCOA: ***C**hip-**O**n-**C**hip technology to **O**pen new **A**pplications*
  STMicroelectronics + 19 partners (ams AG,…)
- ESiP: ***E**fficient Silicon Multi-Chip **S**ystem-**i**n-**P**ackage Integration – Reliability, Failure Analysis and Test,*
  Infineon Technologies AG + 41 partners (ams AG,…)

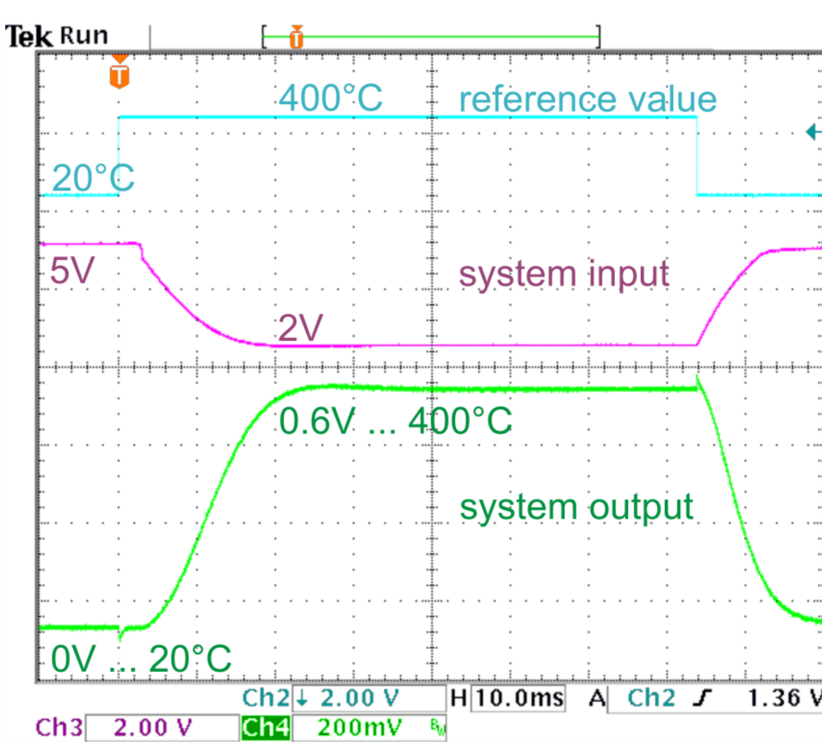


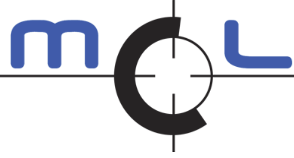


# CMOS microhotplates

- Plate heater: 70 x 70 µm²
- Power consumption at 400°C: 17 mWatt
- Temperature uniformity: 5K, 1K (heat spreader)
- Rise time / Fall time: 12 msec / 25 msec

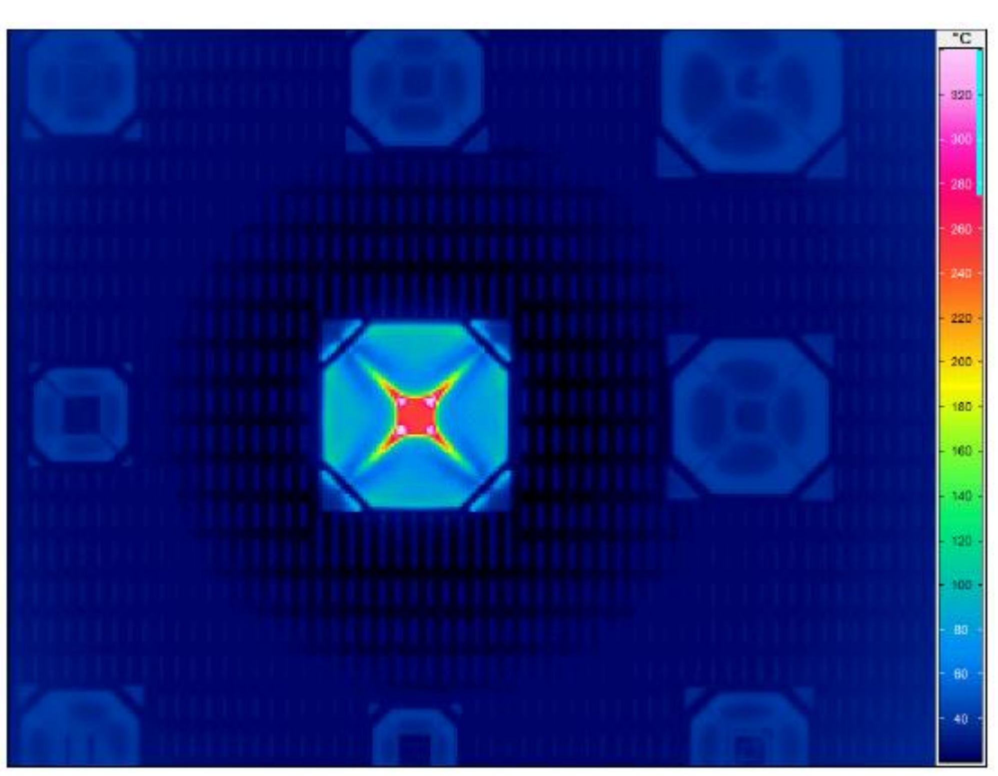


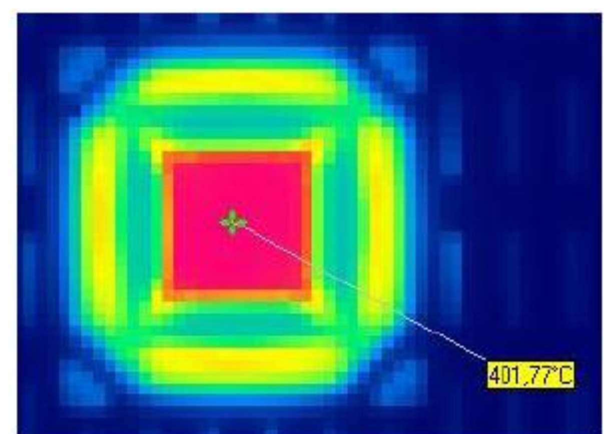


# Thermography measurements

Collaboration: FHWN & VUT

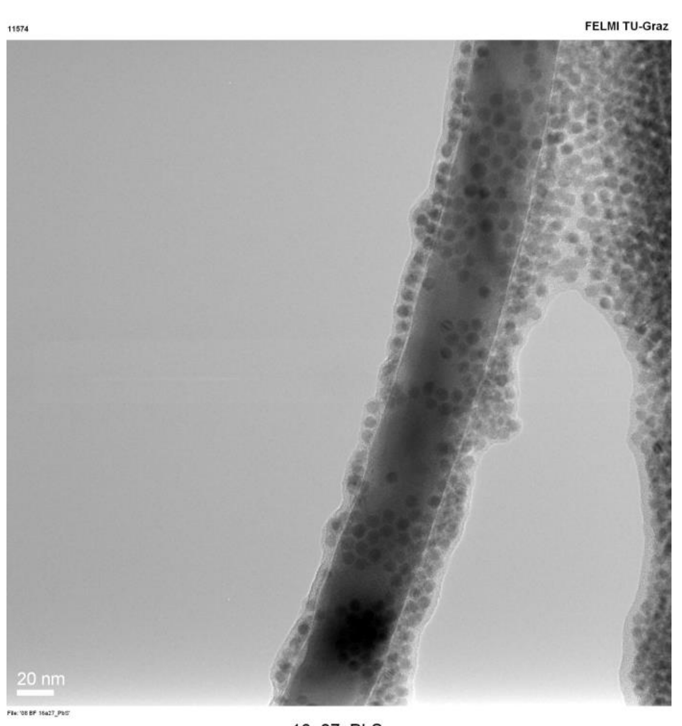



vgl. µHP#2 shows:
Heatspreader flattens temperature distribution

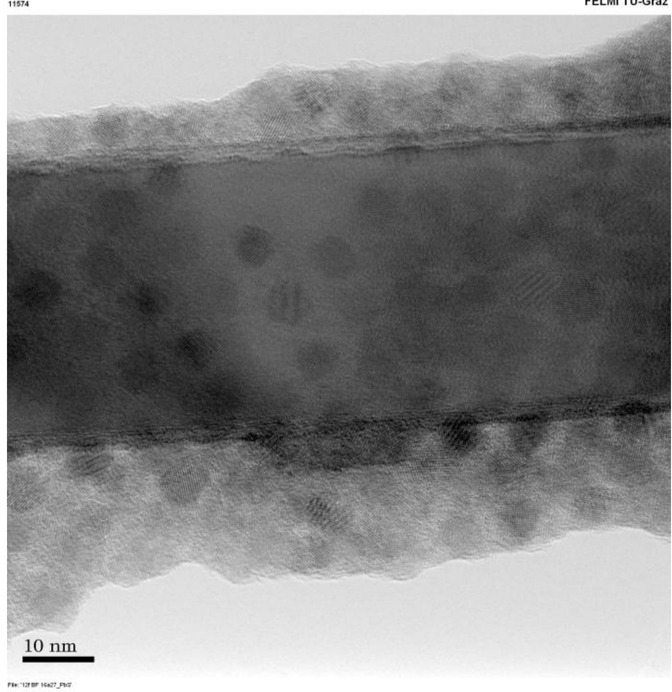


# Selectivity issues

- Functionalization with proper NPs
- Au, AuPt, PtPd, $BaCO_3$,…
- Different operating temperatures
- Dynamic measurements




16a27_PbS
TEM Hellfeld
Serie 08




16a27_PbS
TEM Hellfeld
Serie 12