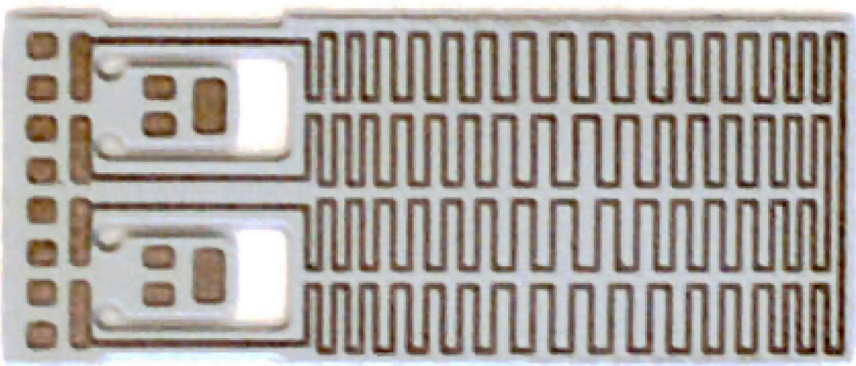
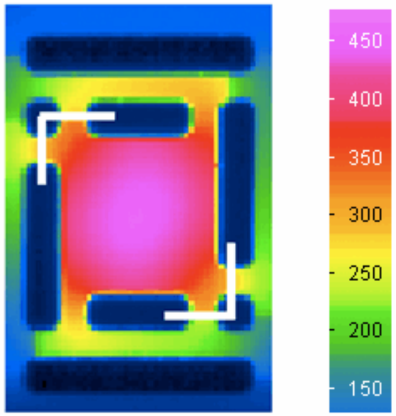
ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

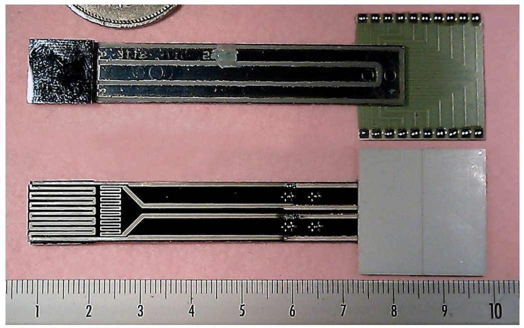
Ceramic cantilever hotplates for devices and testing platforms

Thomas Maeder, Bo Jiang, Fabrizio Vecchio, Conor Slater and Peter Ryser

EPFL-LPM *Laboratory for Micro-engineering for Manufacturing*







Outline

1. Introduction - materials
2. Ceramic hotplates - basics
3. Fuel cells: carriers for MEMS μ-SOFCs
4. Compact hotplates for gas sensing & IR
5. Simple alumina beams for materials testing
6. Conclusions & outlook

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EPFL - Swiss Federal Institute of Technology

ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

EPFL
École Polytechnique Fédérale de Lausanne

One of the two polytechnical schools in Switzerland

10’000	Population on the campus
6’000	Students
3’500	Collaborators
1’400	PhD. Students
250	Professors
550	MSFr budget / year
70	Companies on site
10	New start-ups / year

IMT – Microtechnology - Locations

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The new IMT: 1 Institute on 2 Sites

Lausanne + Neuchâtel Campus

Distance: ~70 km or 45 min

Close collaborations

csem

Hes·so
Haute Ecole Spécialisée de Suisse occidentale

Industry

http://imt.epfl.ch

Topics of LPM thick-film group @ EPFL

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Harsh Environments

Aerospace - Implantable systems
Chemistry – Nuclear - Reactive materials
High-temperature processes

Load sensors

Force / pressure sensing
Integration in packages
Structuration
Medecine / rehabilitation

Technologies

LTCC
Thick-film

Fundamentals

Materials science
Processing
Theory + modelling

Ceramic microfluidics

Microreactors / calorimeters
Gas sensors
Fuel cells

Advanced Packaging

MEMS – Integrated functions
Hermetic – temperature control
Integrated sensors/actuators/fluidics
Structuration – sacrificial layers

Thick-film technology - introduction

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- **Thick-film circuit : series of layers**
  - Screen-printing of layers with a mask
  - Direct dispensing (prototypes)

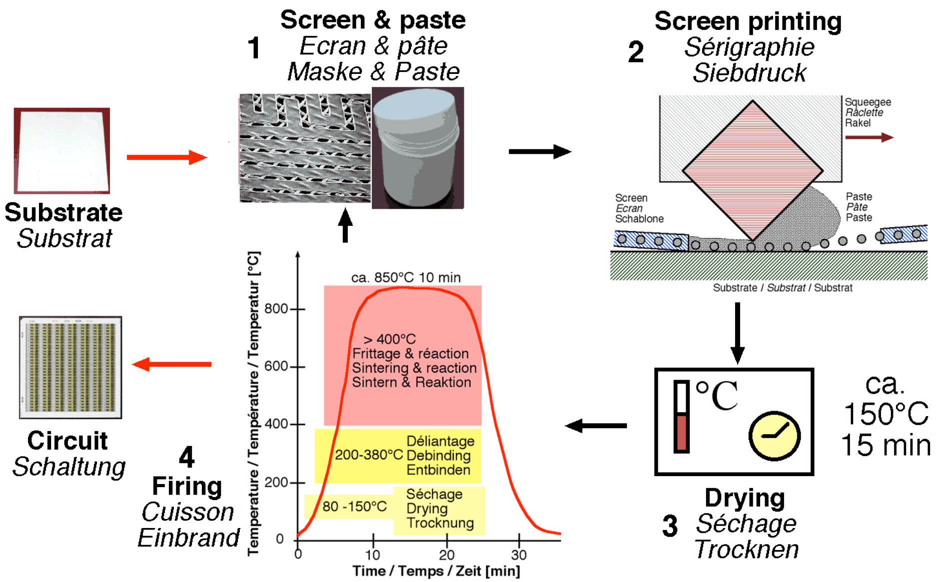
- **Each layer comes as a paste:**
  - Functional material (as powder)
  - Organic vehicle: binder + solvent

- **Materials**
  - Conductors
  - Resistors
  - Dielectrics
  - **…and more!**

Two main routes - process

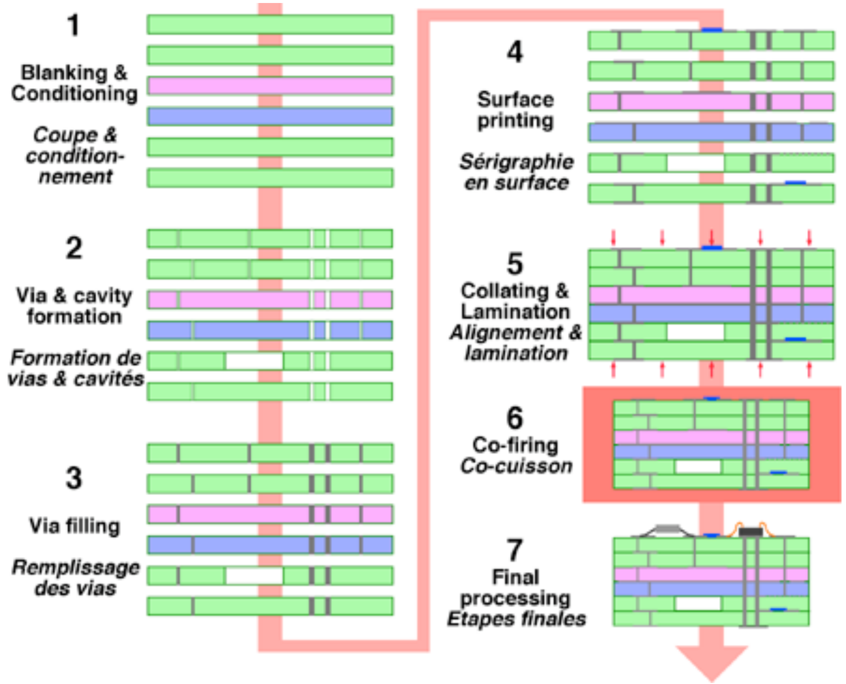
Classical thick-film

- Simple, low-cost
- Complex structuration of substrate impractical
- Mainly cantilevers & bridges



LTCC

- Very good structurability
- Intricate fluidic structures
- Good thermomechanical decoupling in compact layout



Two main routes – typical applications

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Classical thick-film

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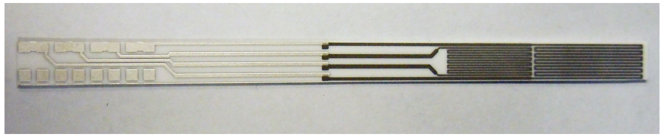
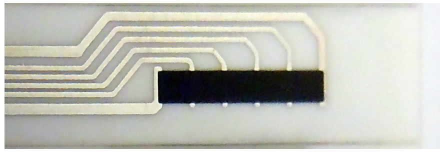
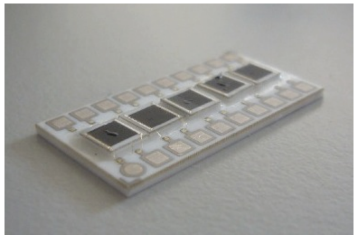




Hotplates for high-temp. materials testing

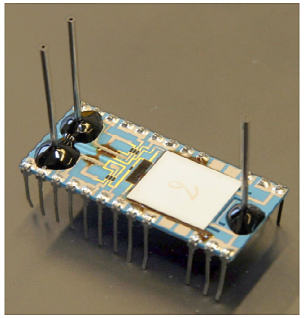
MEMS μ-SOFC reformer thermofluidic test platform



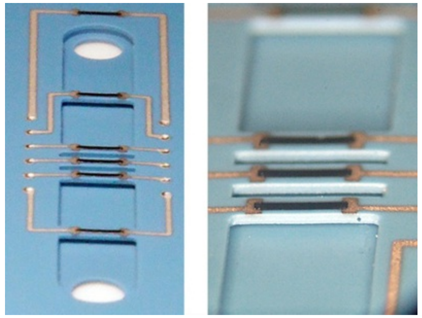
Dummy power components for reliability testing

LTCC

- Very good structurability
- Intricate fluidic structures
- Good thermomechanical decoupling in compact layout

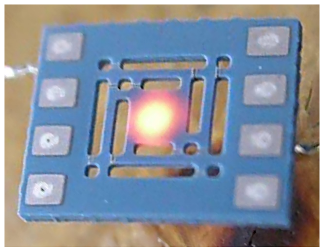


Microreactor / calorimeter

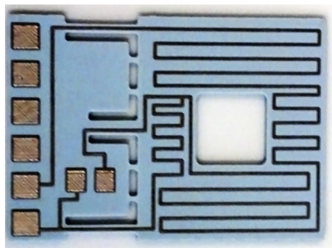


Flow / pressure / temp. multisensor

IR source



Atomic clock T control



Ceramic hotplates – substrate materials

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Approximate properties

T_{max} = max. use temperature
CTE = coefficient of thermal expansion
k = thermal conductivity

Material	T_{max} [°C]	CTE [ppm/K]	k [W/m/K]	Notes
Soda-lime glass	520	8.5	1.0	Standard window glass
"3.3" lab glass	560	3.3	1.2	Sodium borosilicate (Pyrex, Duran, …)
Aluminoborosilicate gl.	730	3.2	1.2	Schott AF32 grade (alcali-free)
Silica glass	>1000	<1	1.3	Pure or almost pure SiO_2
LTCC (DP 951)	580	~6	~3	Very common LTCC material
LTCC (Her CT800) LTCC (CT GC)	>750	~6	~3-4	More crystallising LTCCs
Alumina (Al_2O_3 96%)	>1000	~7	~20	Standard ceramic substrate
Zirconia (stabilised)	>1000	~11	~3	Strength, ionic conductor

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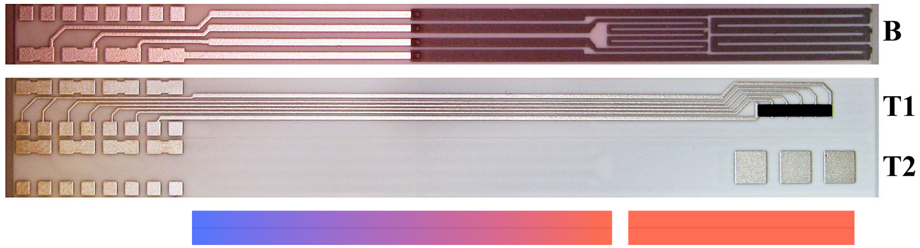
Thermomechanical decoupling

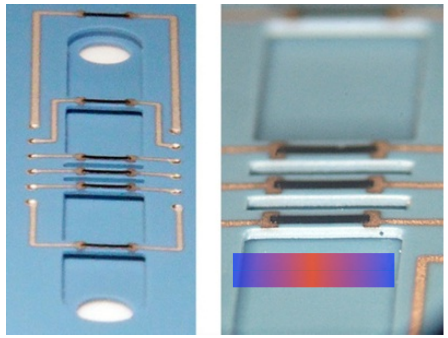
- **Thermal decoupling**
  - Slender bridge sections and/or structuration
  - Low thermal conductivity
  - Convection & radiation important (dominant!)

- **Mechanical decoupling**
  - Allow for thermal expansion
  - Strain -> movement
  - Slender, flexible bridges
  - Process issues!

Materials testing

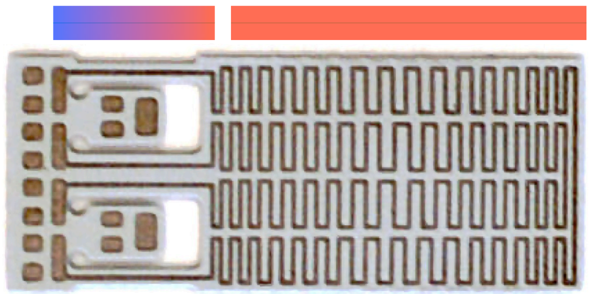
L ~ 50 mm
b = 7 mm
h = 250…500 *µ*m





Gas multisensor

L ~ 2-3 mm
b = 7 mm
h = 100…200 *µ*m



***µ*-SOFC platform**

L ~ 7 mm
b ~ 2 mm (total)
H ~ 1 mm (fluidics)

Thermomechanical decoupling

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- **Thermal decoupling**
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  - Convection & radiation important (dominant!)

Class	***k*** [W/m/K]
Glasses	0.5…1.5
LTCC	3…5
Zirconia (stabilised)	~3
Alumina (Al_2O_3 96%)	15…25

- **Mechanical decoupling**
  - Allow for thermal expansion
  - Strain -> movement
  - Slender, flexible bridges
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Ranking (heat conduction)

- Best: LTCC (structuration)
- Good: glass & ZrO_2
- Less optimal: Al_2O_3

Thermomechanical decoupling

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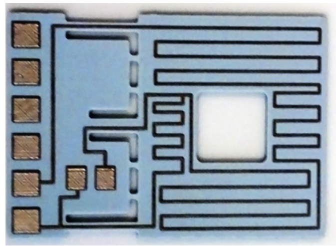
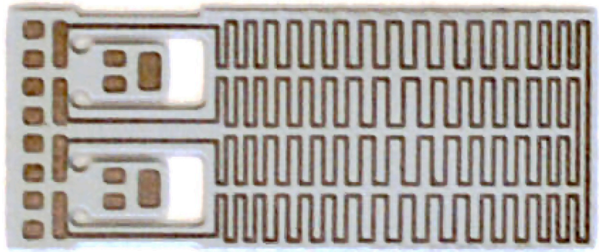
Atomic clock T control



T_{cold} ~ 25°C
T_{hot} ~ 80°C

W_{cond} ~ 1'000 K/W
$W_{observed}$ ~ 80 K/W (in free air)

μ-SOFC platform



T_{cold} ~ 50°C
T_{hot} ~ 550°C

W_{cond} ~ 400 K/W
$W_{observed}$ < 100 K/W (in insulated oven)

Countermeasures

- Insulation
- Radiation shields
- Vacuum

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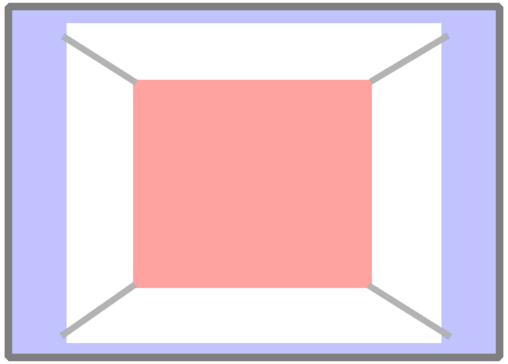
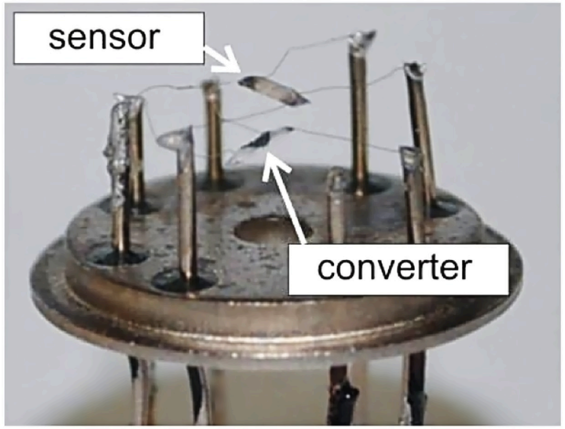
Suspended by bond wires (common in classical gas sensors)





2011 Shaposhnik

Good properties
Cumbersome process…

Thermomechanical decoupling

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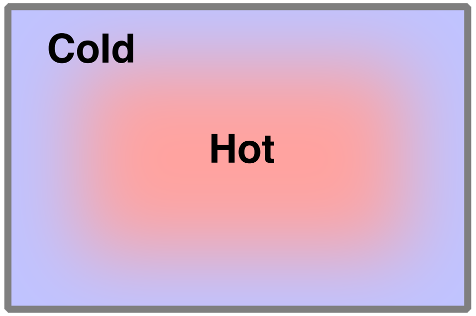

No structuration

Facing bridges

- **Mechanical decoupling**
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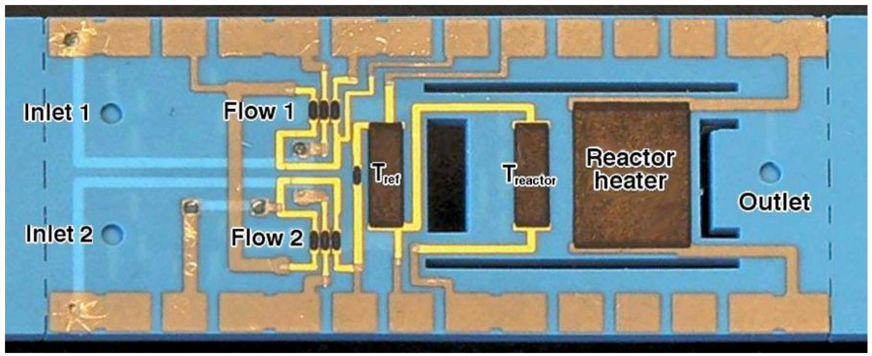

Thermal stresses!
Only OK for small ΔT

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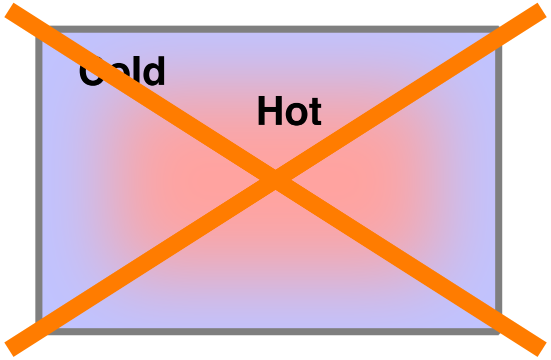
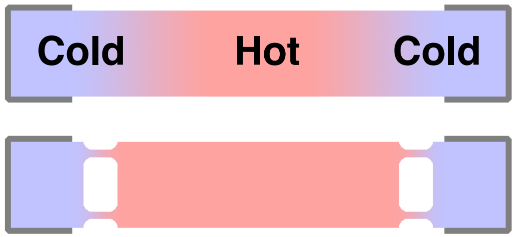


No structuration



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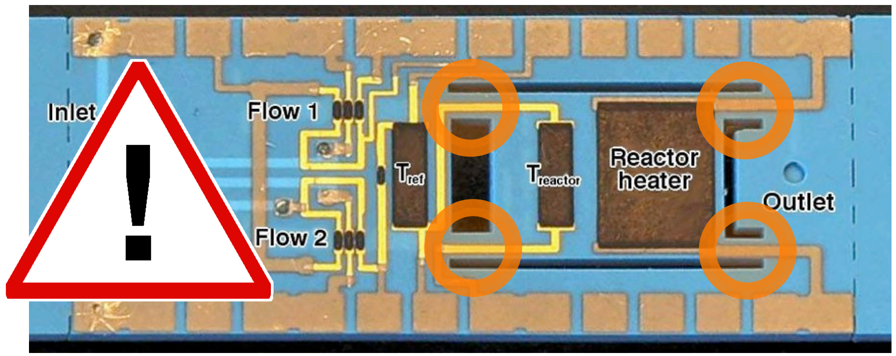

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Cantilevers

- **Mechanical decoupling**
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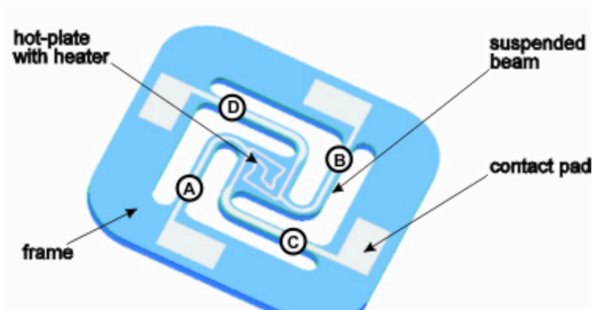
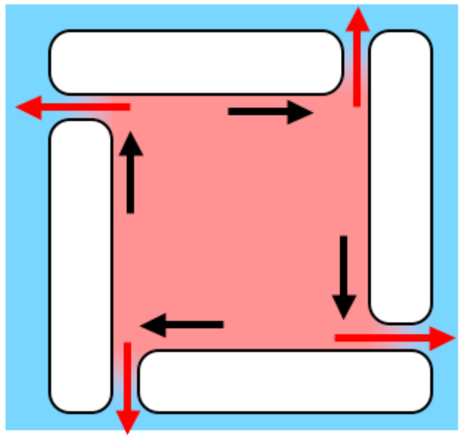
  - Slender, flexible bridges
  - Process issues!

Structured hotplate



2005 Kita



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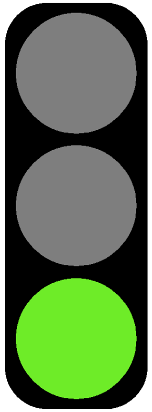
Cantilevers

OK



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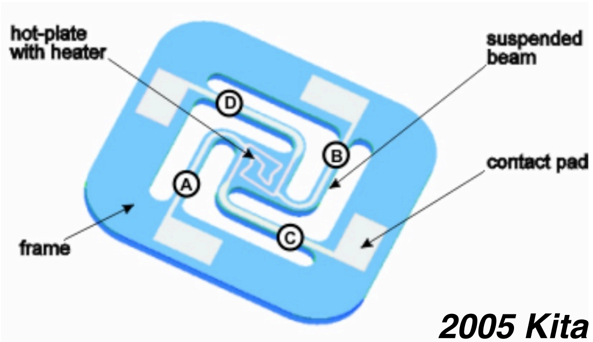
Structured hotplate



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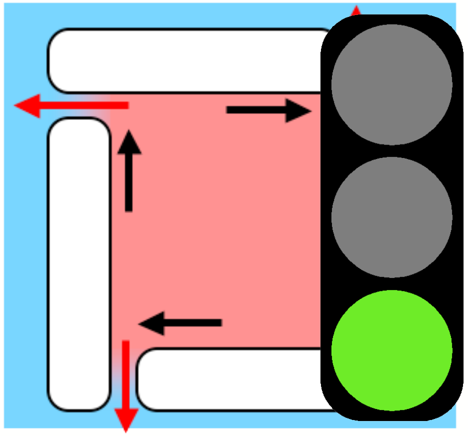

OK

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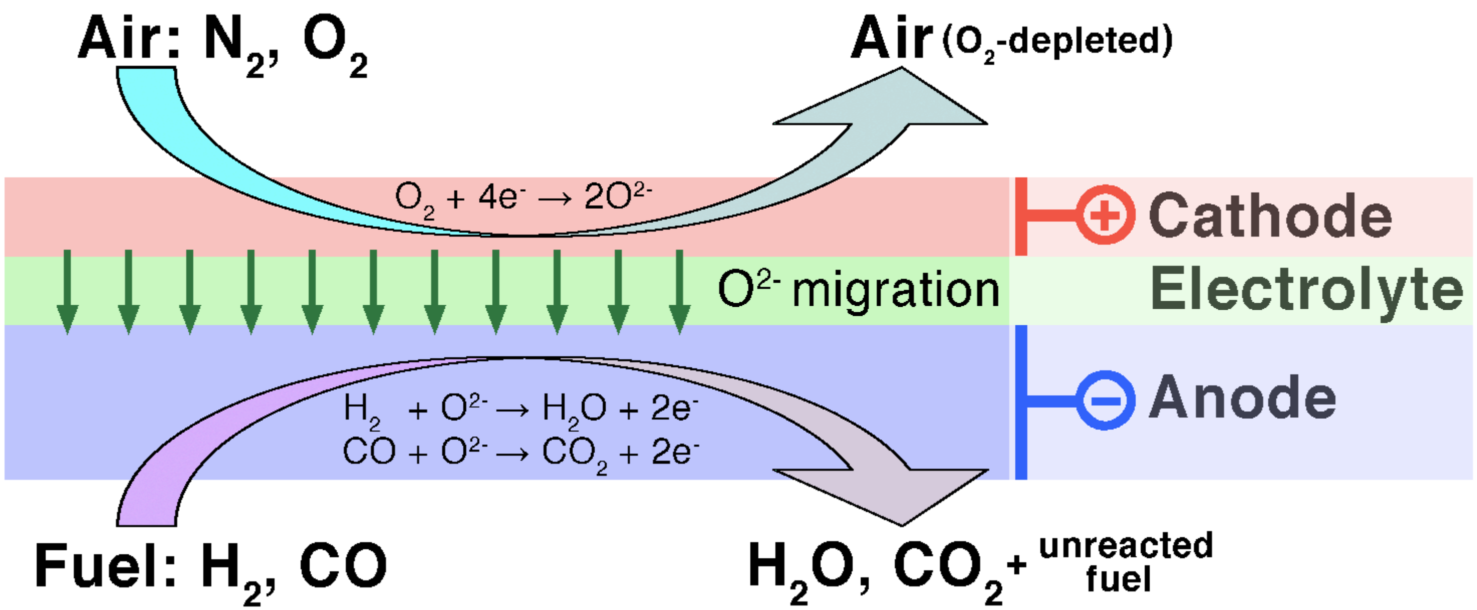
SOFC features

- No liquid phase, no wetting required
- Relatively insensitive to pollutants
- Compatible with a wide range of fuels

- ***High temperatures, stability issues (≈800°C)***



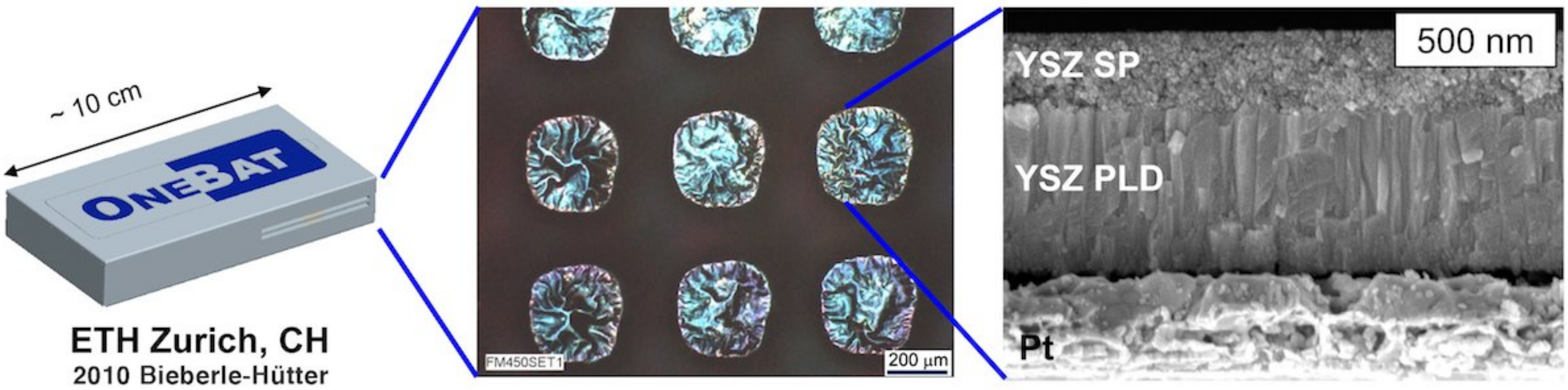
SOFC principle

Downscaling of SOFCs

- Thinner membranes
- High-activity electrodes
- Alternative fabrication techniques

→ **Lower operating temperatures possible**

Higher surface/volume



Module **Membrane array** **Thin-film stack**

http://www.nonmet.mat.ethz.ch/research/onebat

Downscaling of SOFCs - applications

Envisioned application range

Media player: ~1…5 W

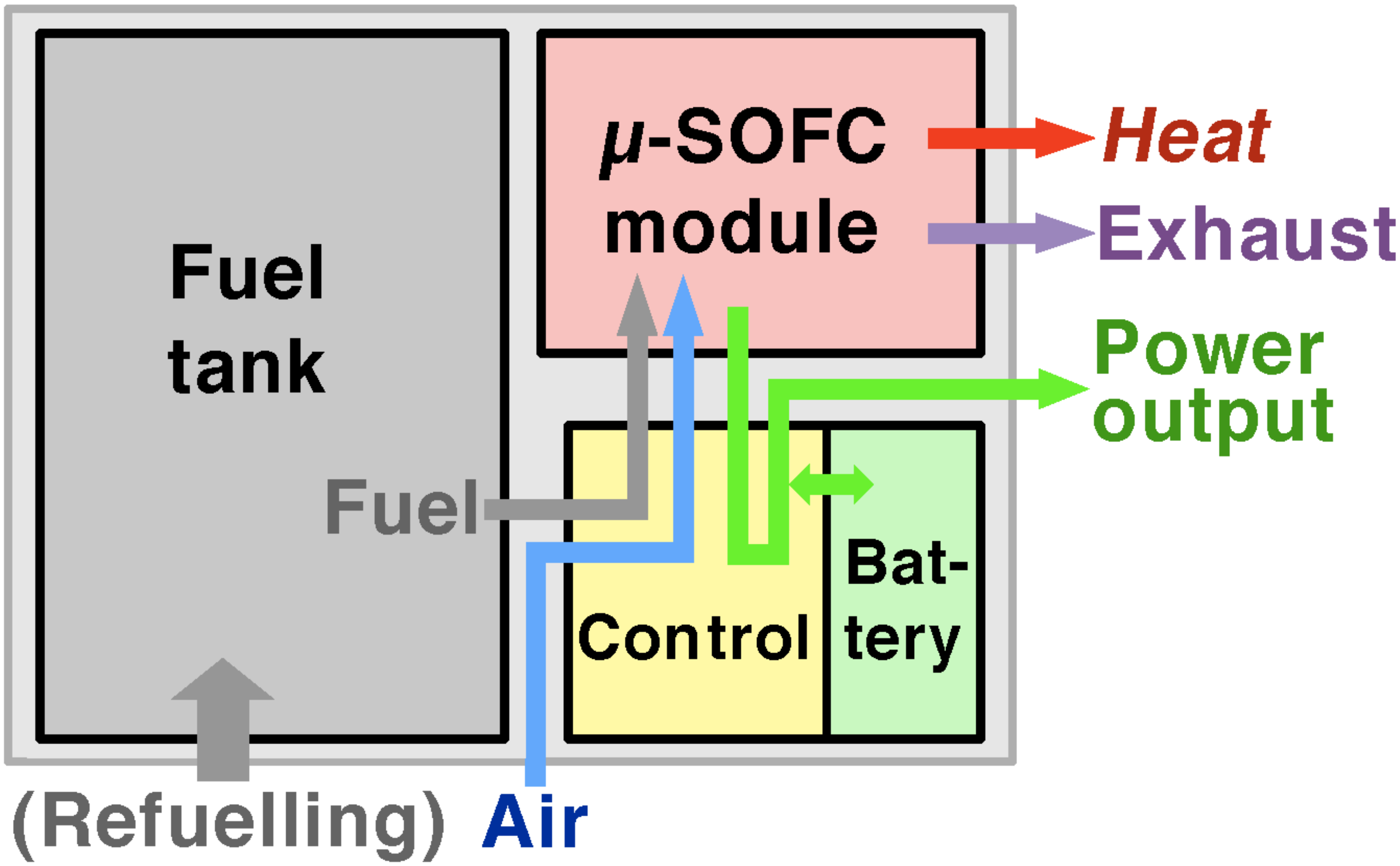
Portable PC : ~50 W

Camcorder : ~10 W

Power requirements

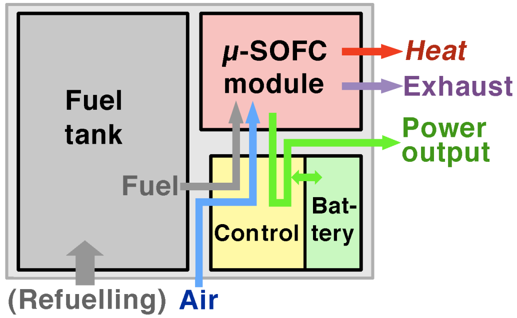
μ-SOFC system principle

- Integration into a single, compact module

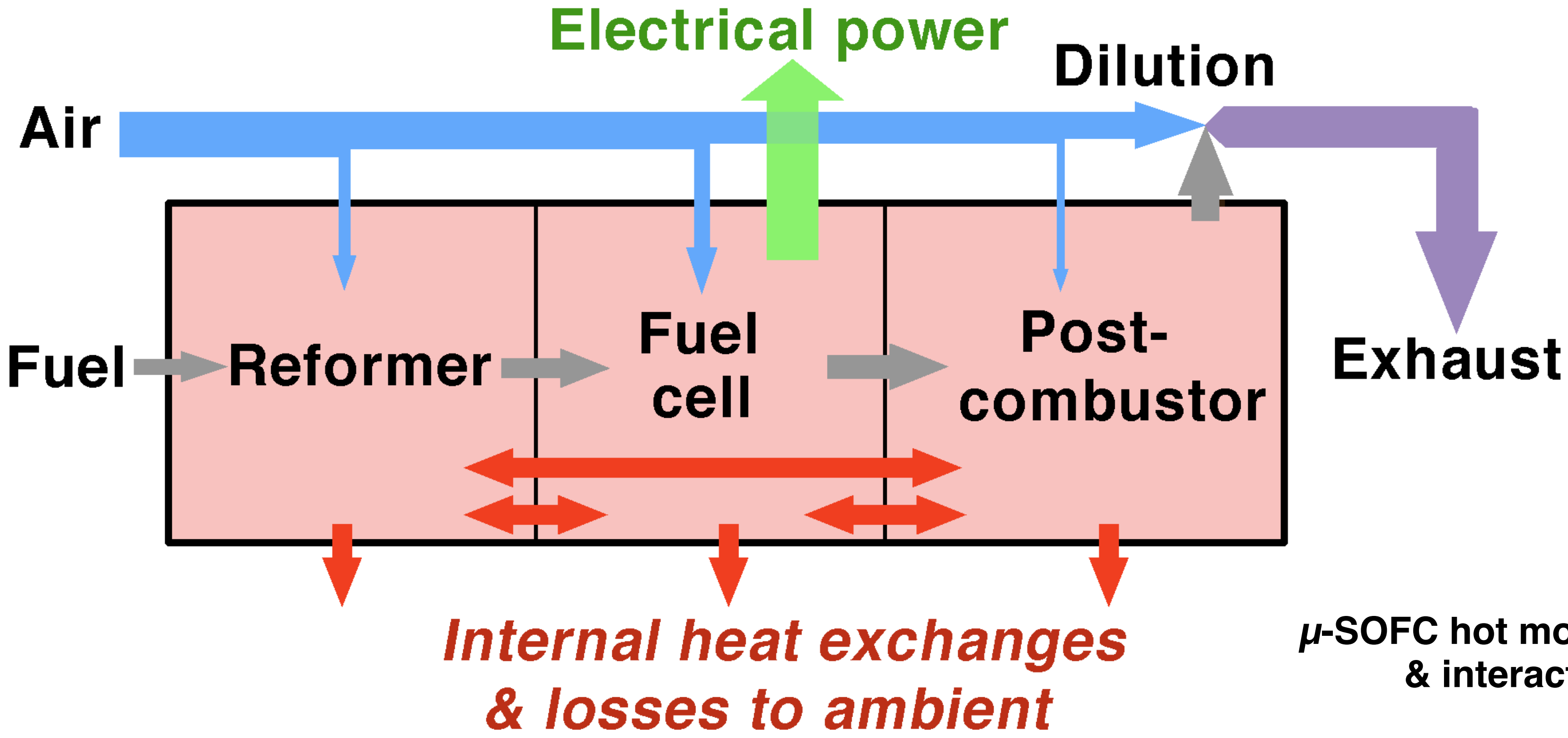


μ-SOFC system issues

- Gas reforming
- Electrochemical conversion
- Post-combustion



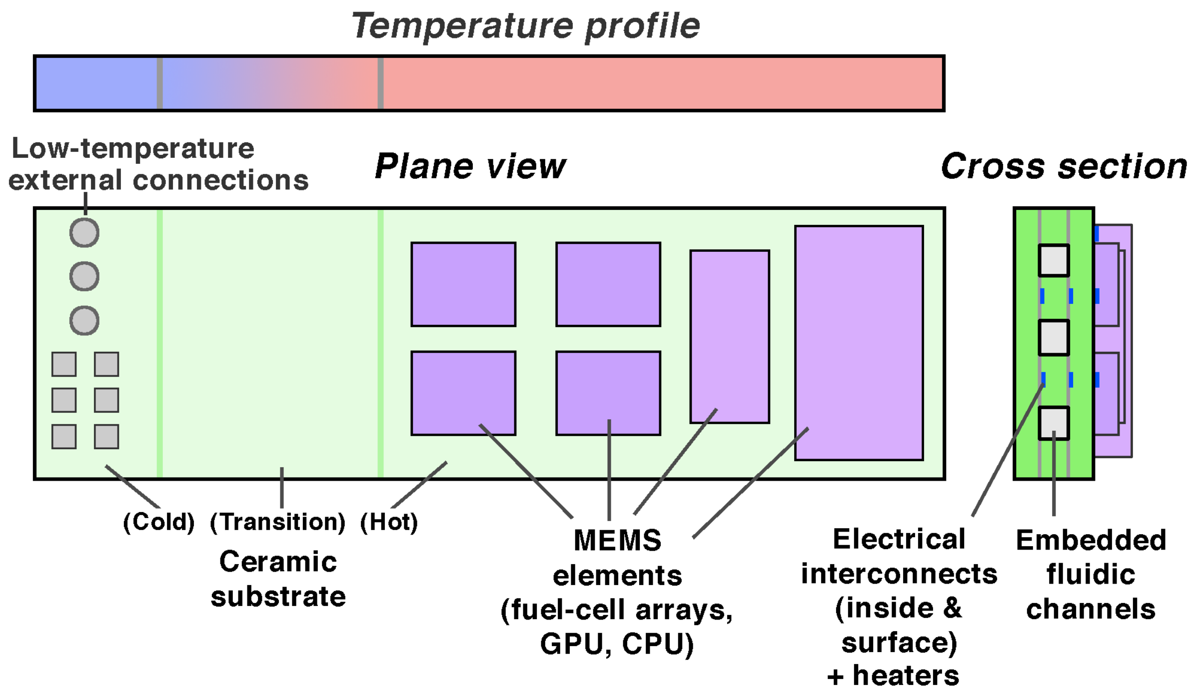
μ-SOFC module principle



μ-SOFC hot module flows & interactions

LTCC μ-SOFC module concept

- Simple "stick" structure
- Facile thermal & mechanical decoupling
- External (electrical & fluidic) connections at low temperature
- Integration of fluidics & possibly gas processing



LTCC μ-SOFC module packaging concept

LTCC *μ*-SOFC module concept

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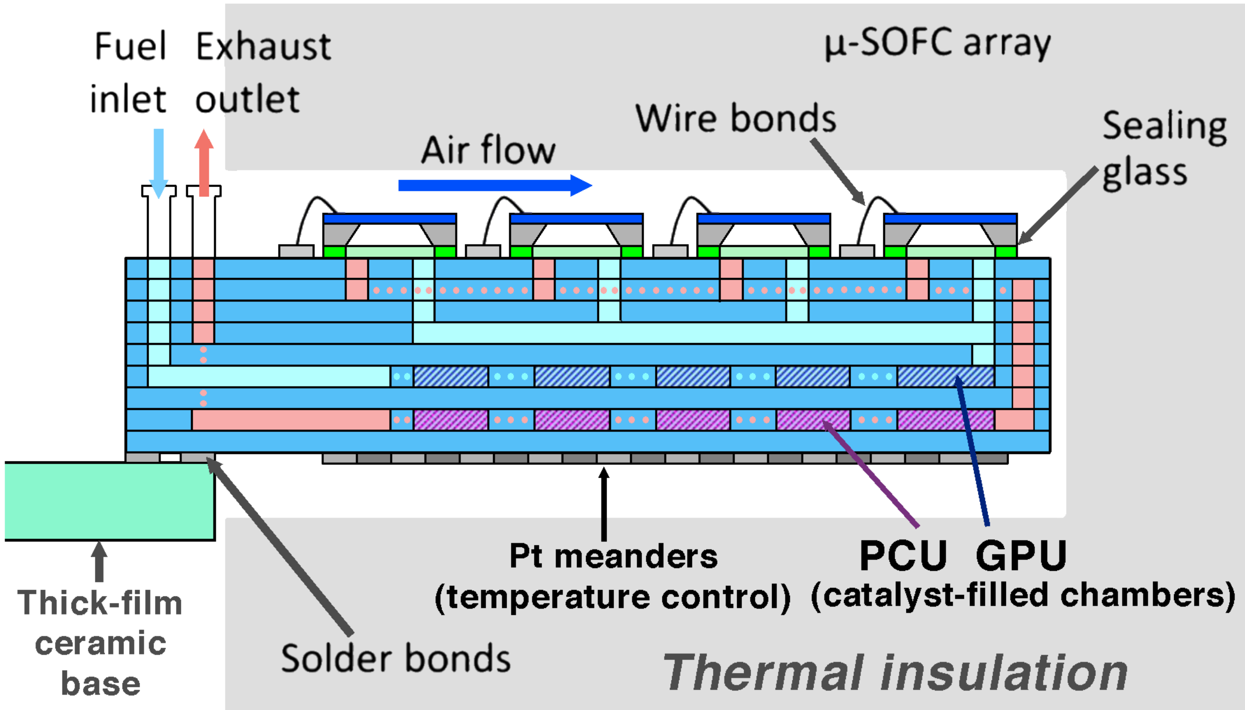
- Facile thermal & mechanical decoupling
- External (electrical & fluidic) connections at low temperature
- Integration of fluidics & possibly gas processing

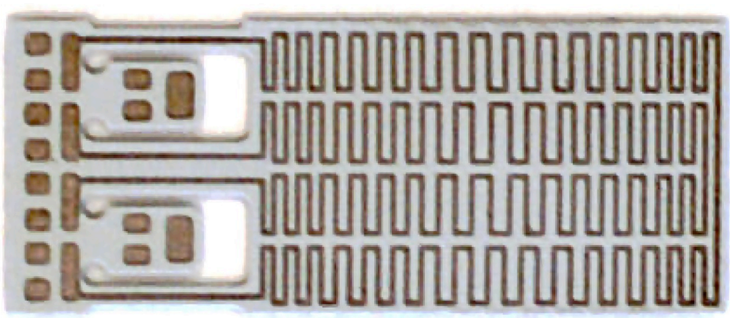


LTCC *μ*-SOFC module

PCU = post-combustor
GPU = reformer

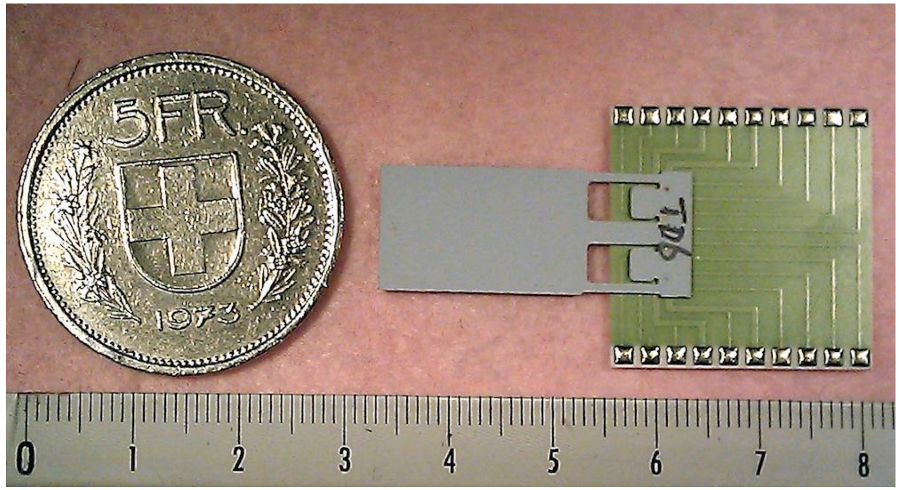
LTCC *µ*-SOFC test platforms

- Thermal decoupling by slender bridges
- Electrical (bottom) & fluidic (top) connections
- Heating & temperature control by Pt meanders
- SMD-soldered to thick-film base for easy connections & cooling of "cold" side
- Compatible with full system



LTCC *µ*-SOFC test platform

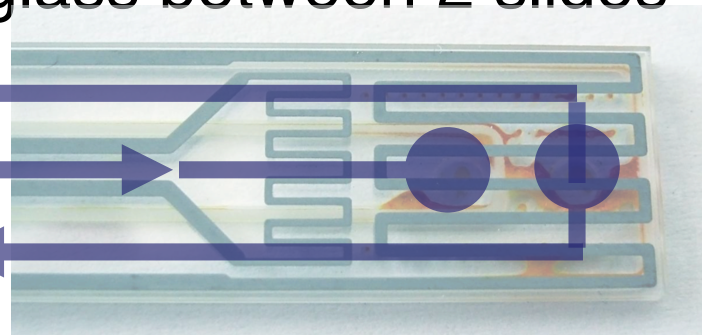
Pt track

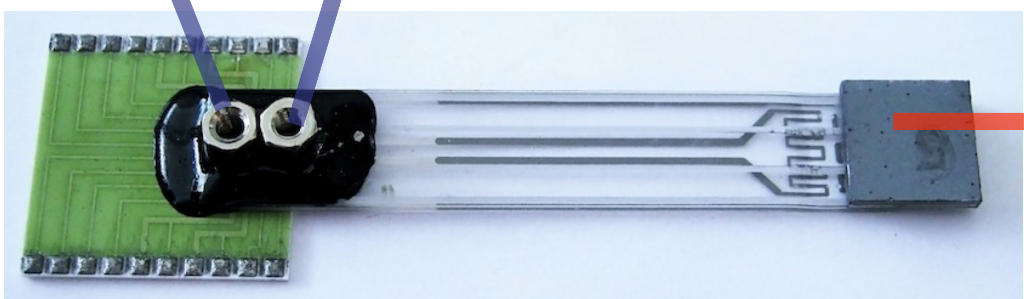


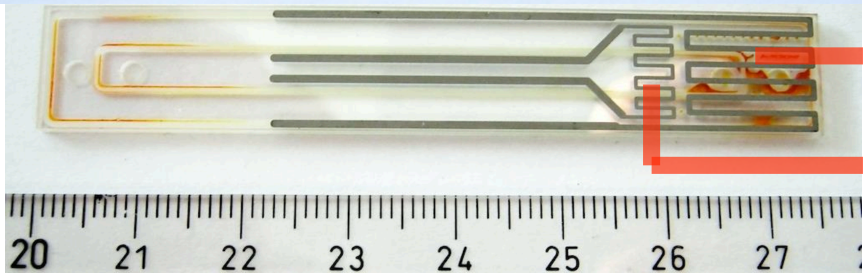
Platform mounted onto base

Simple glass testing platform

- Perfect thermal match with Si MEMS
- For sealing of large structures & component tests
- Fluidics by screen printing of sealing glass between 2 slides
- Compatible with testing jig
- AF32 glass, >700°C

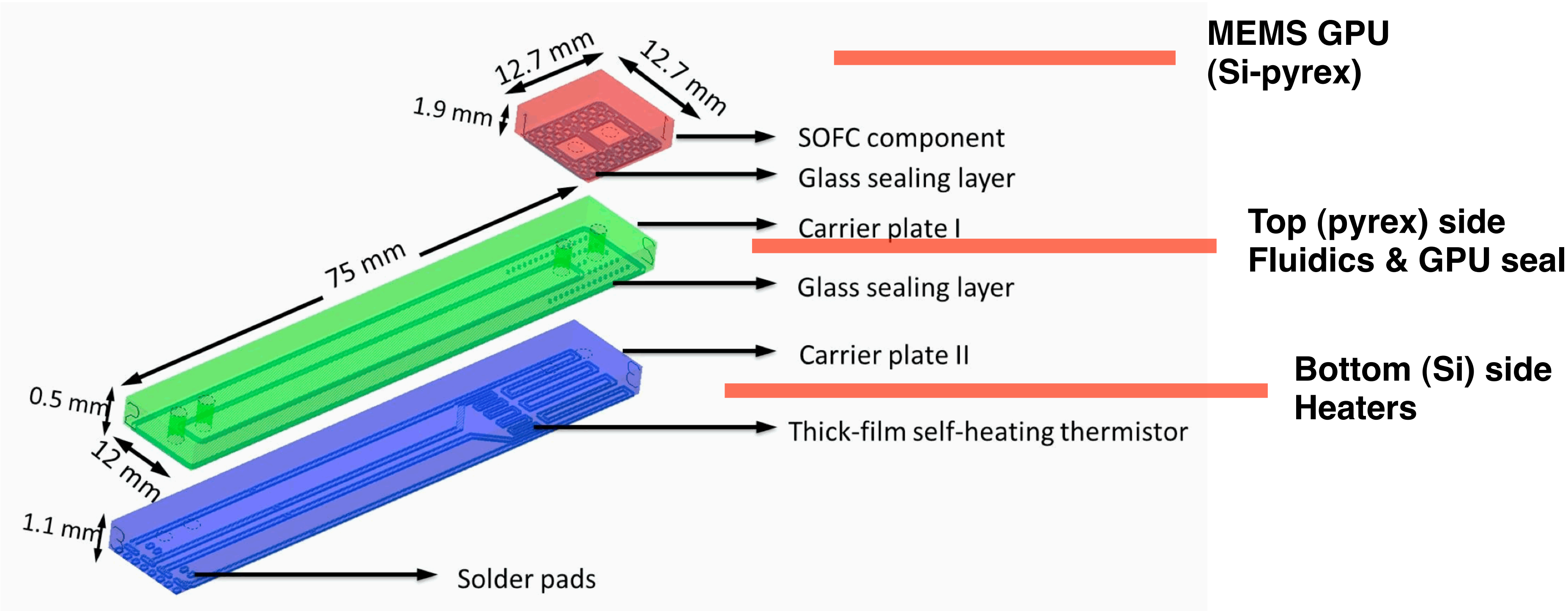






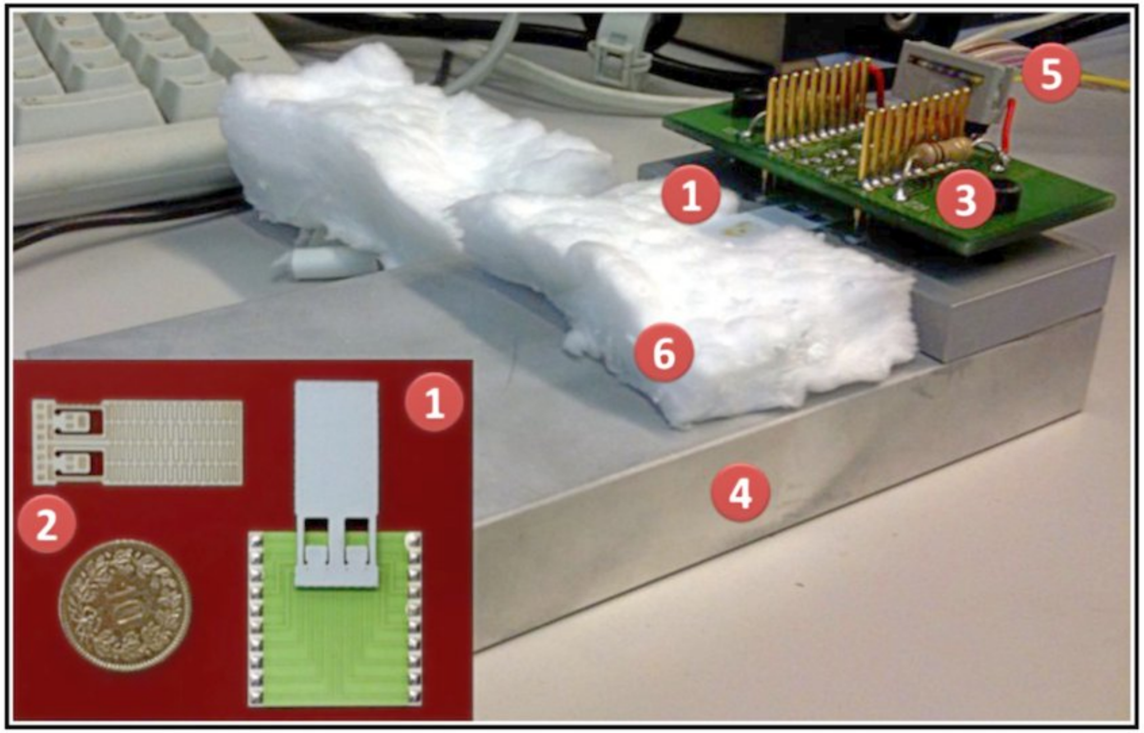
Silicon-glass testing platform

- Perfect thermal match with Si MEMS
- For sealing of large structures & component tests
- Fluidics by screen printing of sealing glass between 2 slides
- Compatible with testing jig
- AF32 glass, >700°C



Testing setup

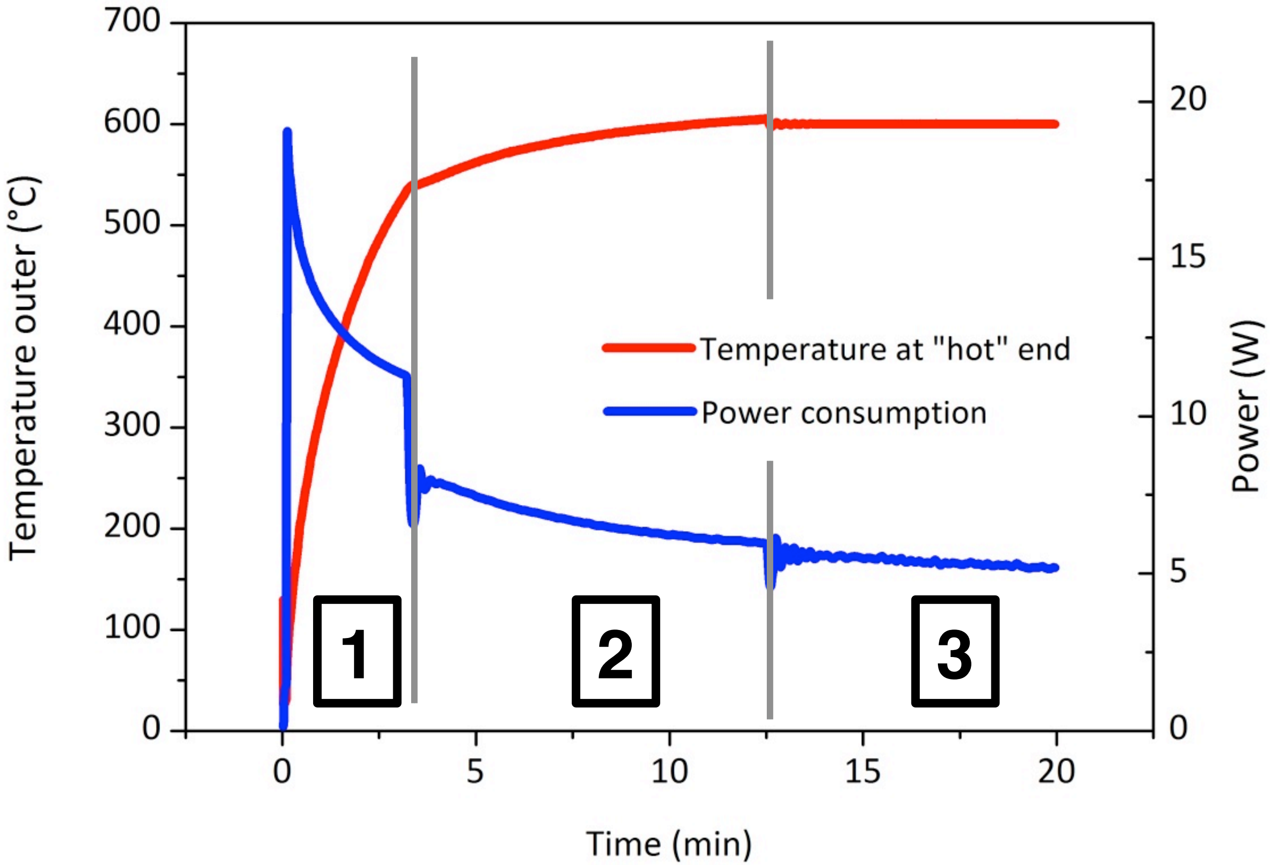
- Modular system
- Extra connections for tested elements
- Standardised layout, compatible with both LTCC & glass



1. Mounted LTCC platform
2. LTCC platform (bottom, Pt tracks)
3. PCB adapter
4. Al base & heatsink
5. External electrical connections
6. Superwool™ electrical insulation

Heating tests (glass carrier)

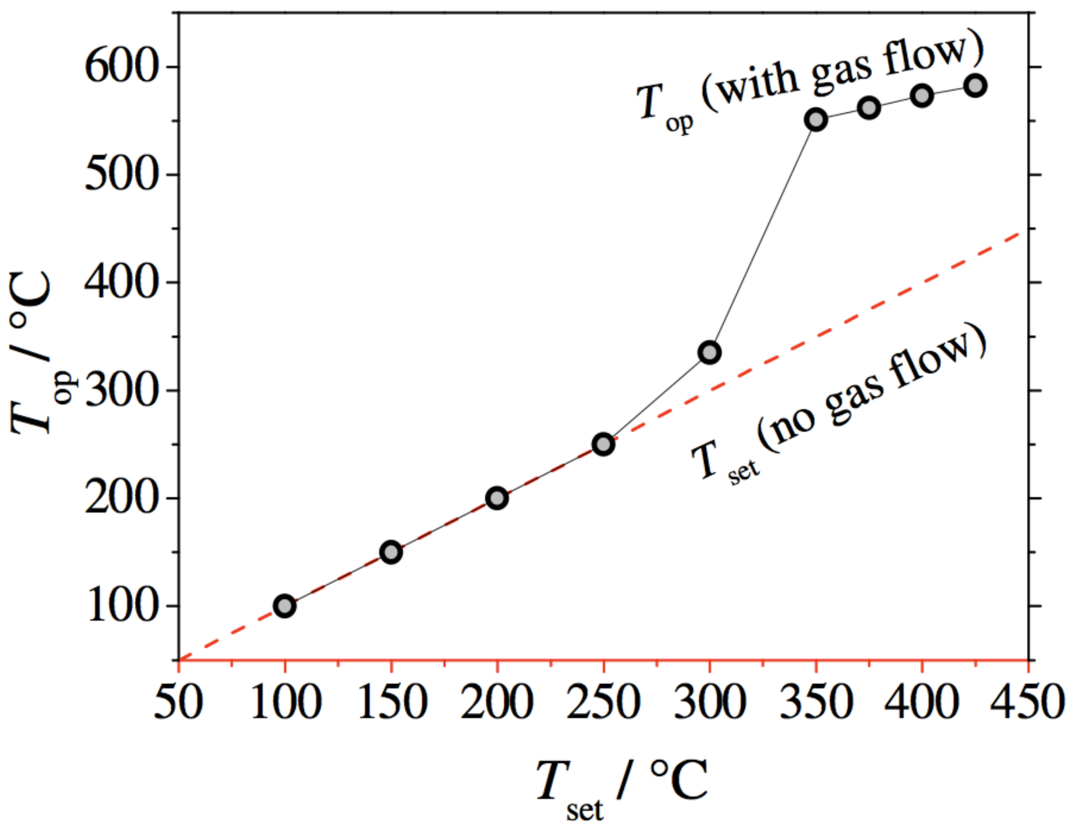
- Heating in furnace, set temperature = 600°C
- Delay due to heating furnace inner side
- Three stages of heating clearly seen



1. Heating at full power, both heaters
2. Guard heater set temperature reached
3. Main heater set temperature reached

Gas reformer test

- Conversion of hydrocarbons to syngas by CPOX (catalytic partial oxidation)
- $C_xH_y + O_2$ -> H_2, CO over catalyst in GPU

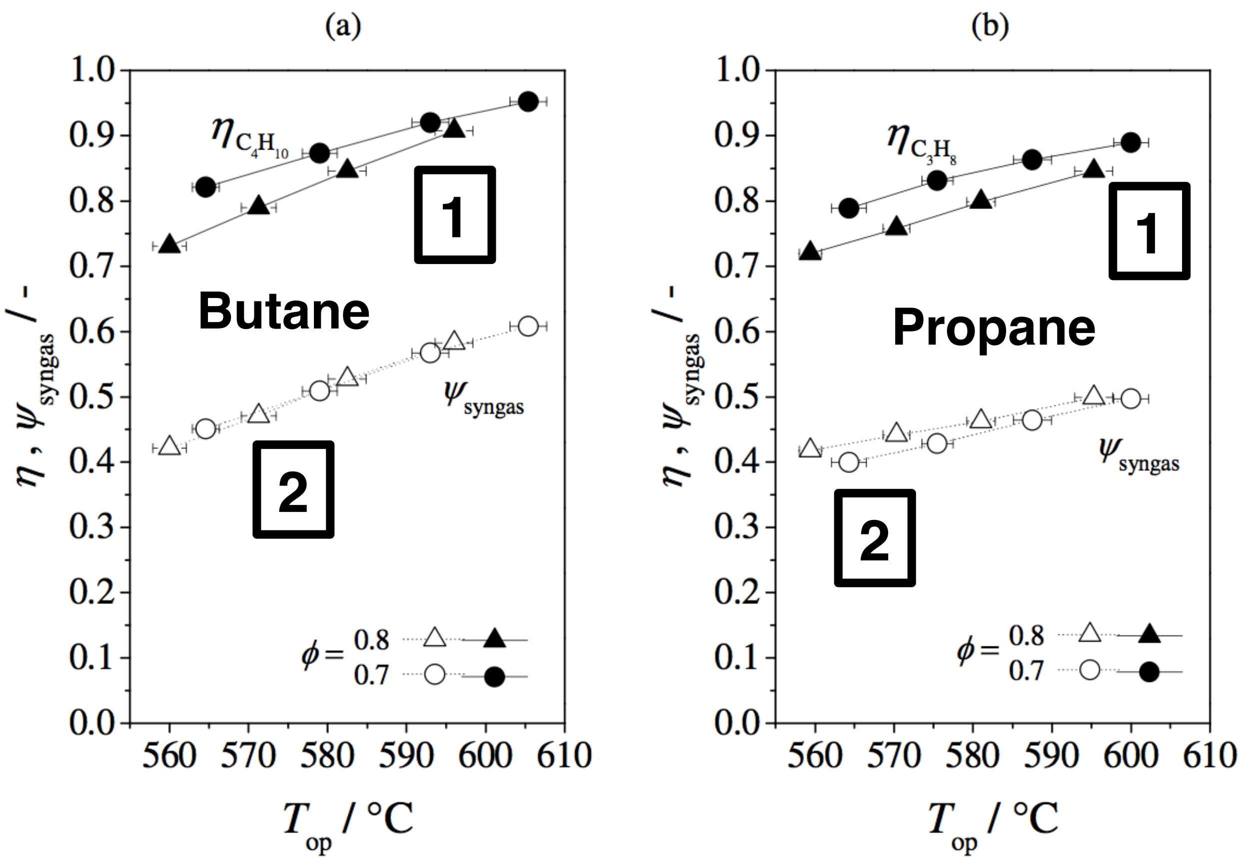


"Light-up" of converter clearly seen above 300°C due to exothermic CPOX reaction

2012 Santis-Alvarez, in press

Gas reformer test

- Conversion of hydrocarbons to syngas by CPOX (catalytic partial oxidation)
- $C_xH_y + O_2$ -> H_2, CO over catalyst in GPU



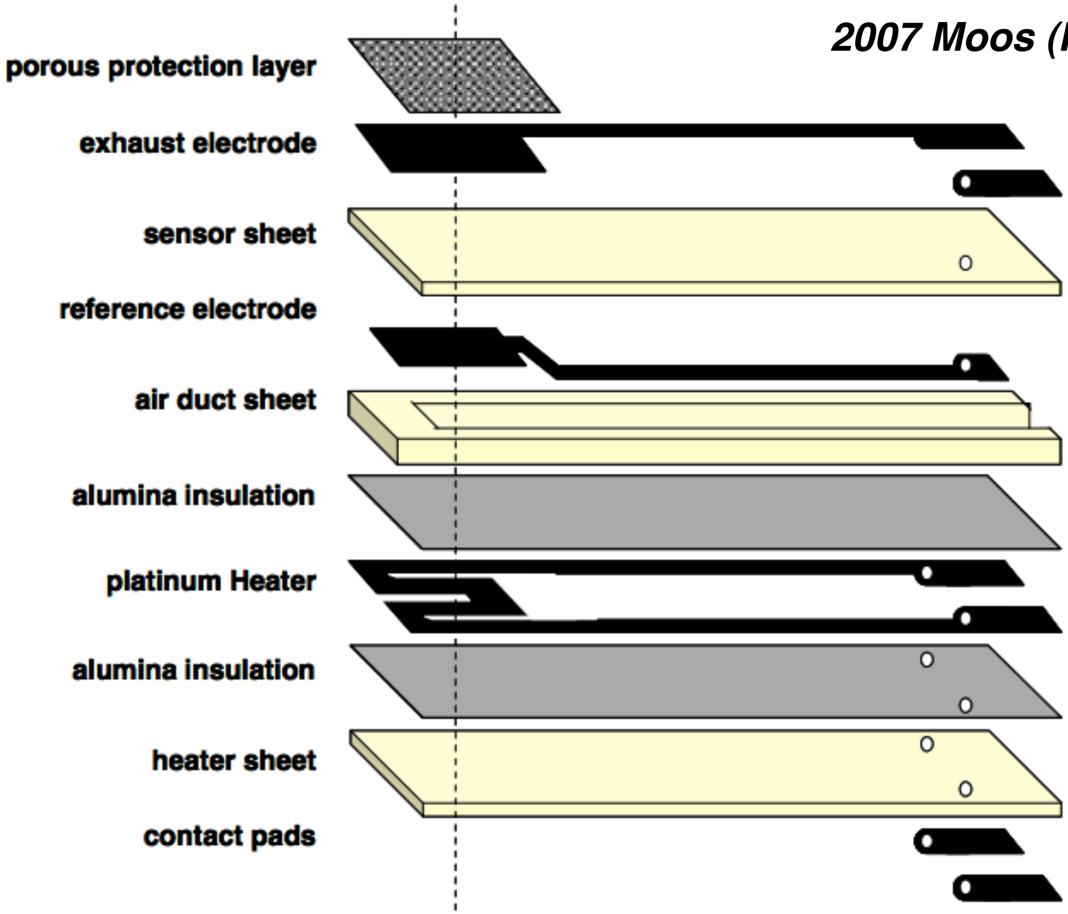
1. Disappearance of propane / butane gas
2. Syngas yield = (H_2+CO) / ideal

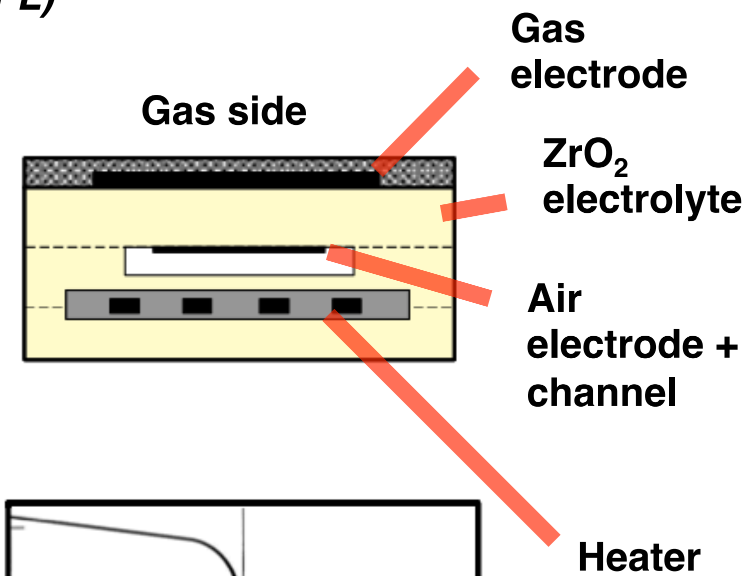
2012 Santis-Alvarez, in press

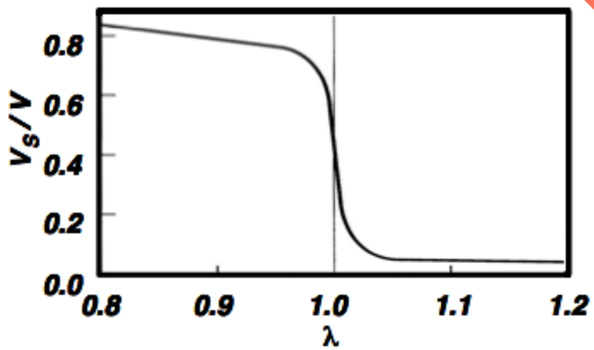
Related: potentiometric oxygen sensor

- Classical: with air duct to outside world
  (air = reference electrode for $p(O_2) \approx 20$ kPa)

2007 Moos (IMAPS-PL)

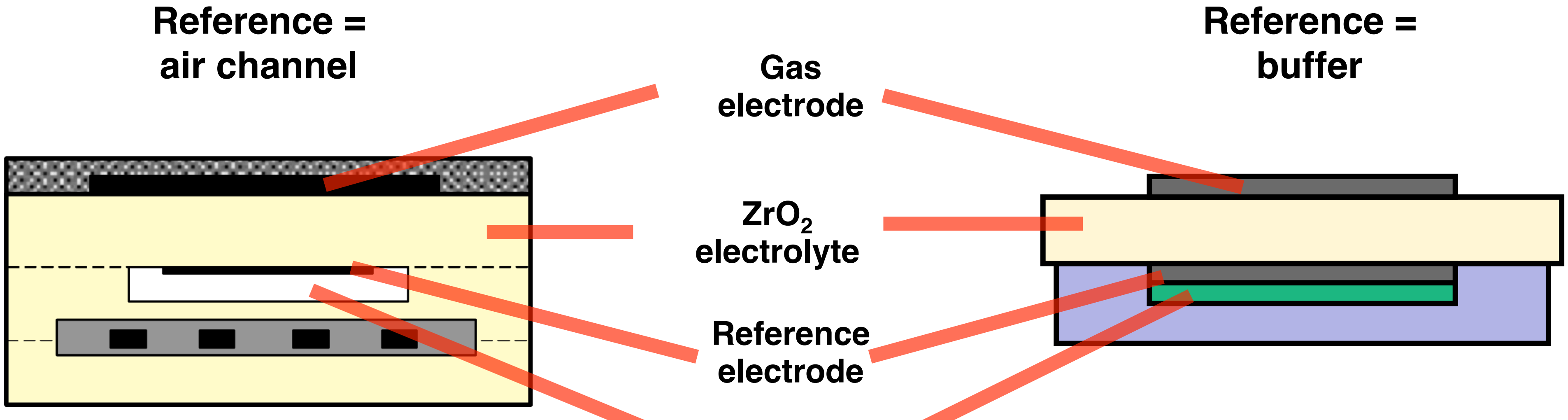






Related: potentiometric oxygen sensor

- Alternate: with oxidoreduction buffer
- Air channel: $p(O_2) \approx 20$ kPa
- Buffer: mixture of oxidised & reduced species: set $p(O_2)$



2007 Moos (IMAPS-PL)

2012 Kobierowska & al. (IMAPS-PL)

Outline

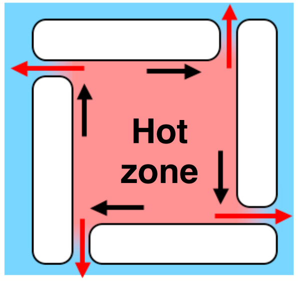
ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

1. Introduction - materials
2. Ceramic hotplates – basics
3. Fuel cells: carriers for MEMS *μ*-SOFCs
4. **Compact hotplates for gas sensing & IR**
5. Simple alumina beams for materials testing
6. Conclusions & outlook

Compact hotplates vs. large ones

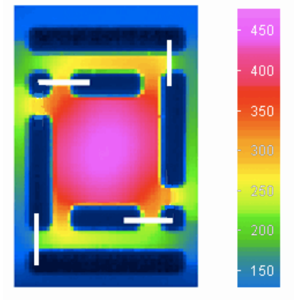
- **Advantages**
  - Small
  - SMD-compatible assembly
  - Easy to fabricate
  - Well-defined mechanics
- **Issues**
  - Poorer resolution than photolithography
  - Not for very small devices
  - Poorer space utilisation than cantilevers
  - Difficult process for very fine bridges
  - Slower than MEMS

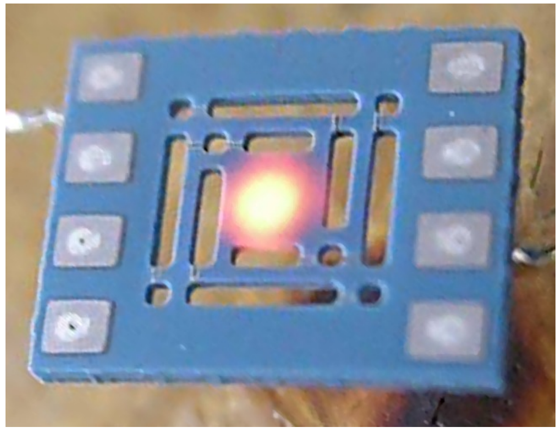
Basic principle

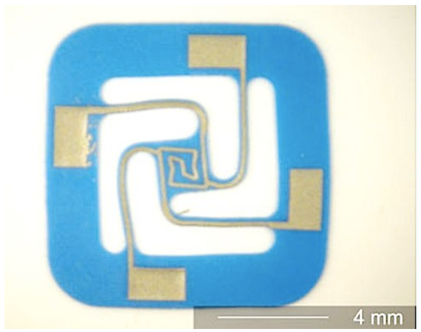


Cold outer frame

Radiant heater







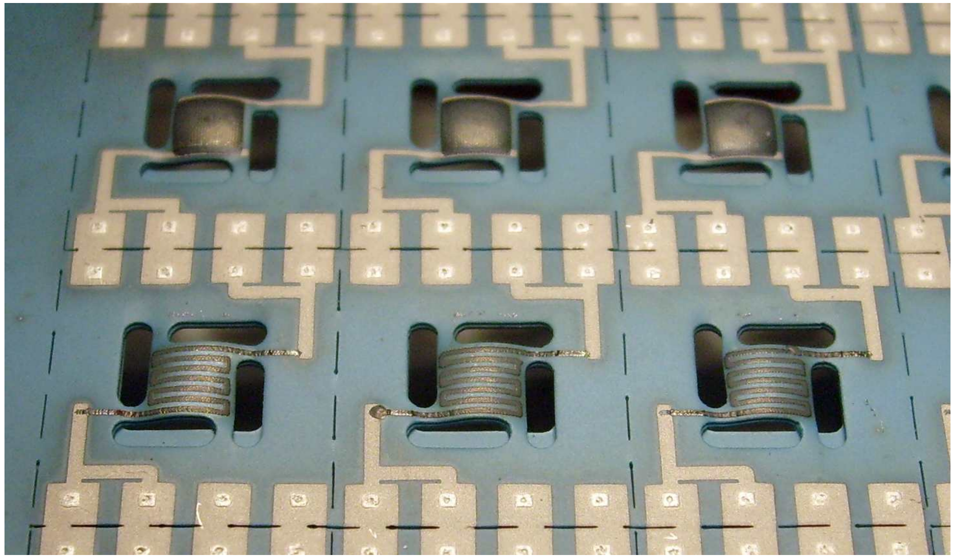
Hotplate for gas sensor

2005 Kita

Compact hotplates – process issues

ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

- **Thermal decoupling**
  - Slender bridge sections and/ or structuration
  - Low thermal conductivity
  - Convection & radiation important (dominant!)



- **Mechanical decoupling**
  - Allow for thermal expansion
  - Strain -> movement
  - Slender, flexible bridges
  - **Process issues!**

- Deformation of thin structures in co-firing
- Difficult to post-print onto thin structures

Compact hotplates – process issues

ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

- **Thermal decoupling**
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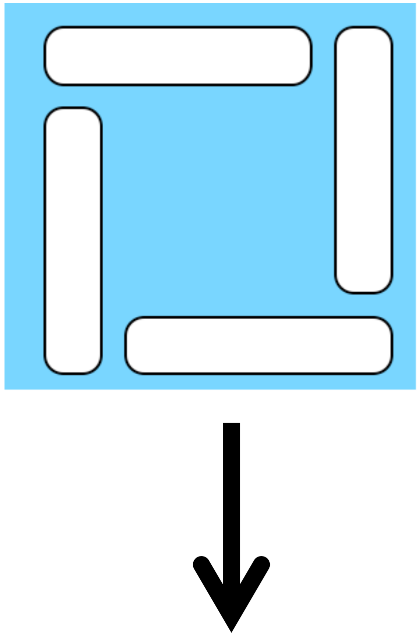
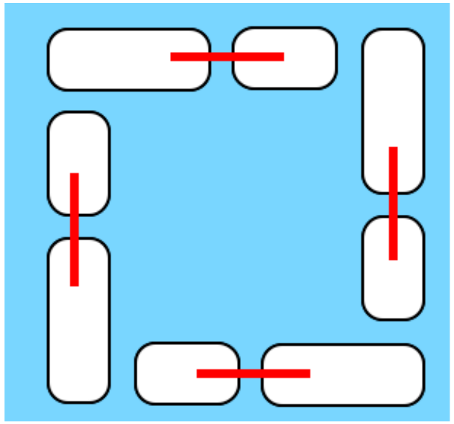
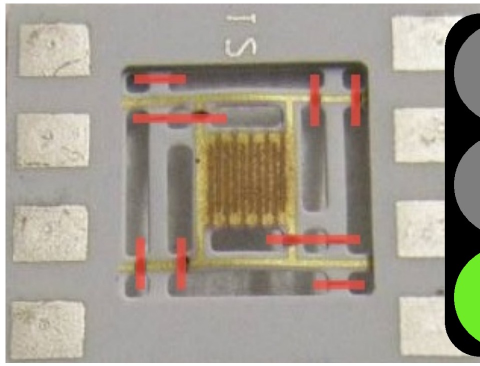


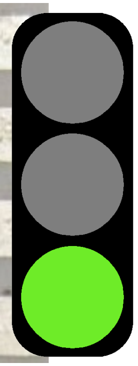
Fully cut out, may break during processing

- **Mechanical decoupling**
  - Allow for thermal expansion
  - Strain -> movement
  - Slender, flexible bridges
  - **Process issues!**

Temporary bridges cut at the end by laser







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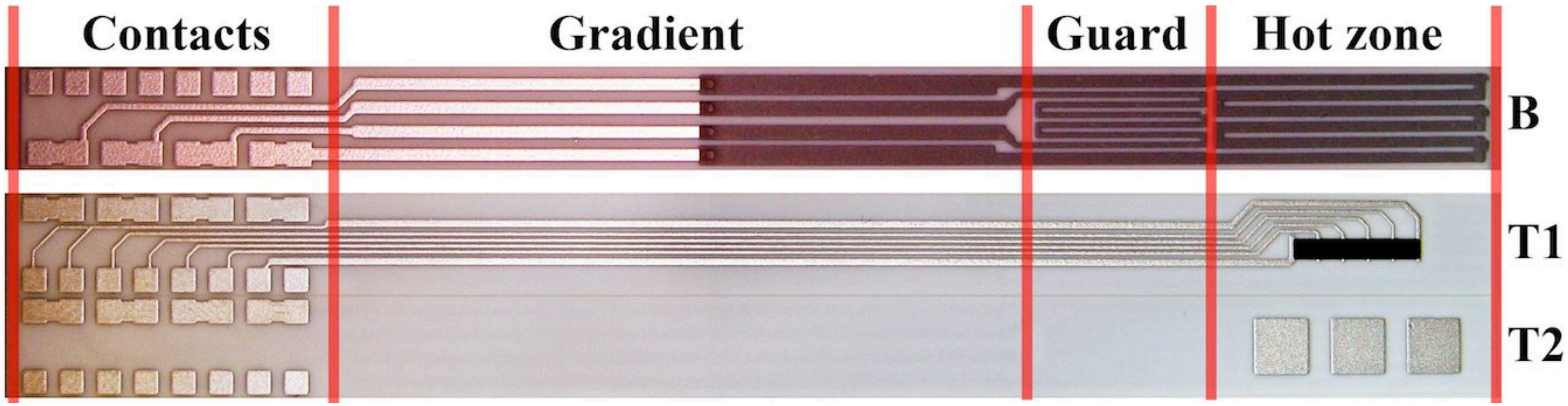
Simple materials testing platform

- **Goals**
  - Test materials to high temperatures
  - Simple, low-cost

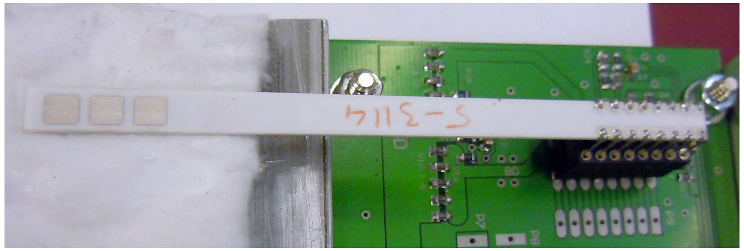
- **Implementation**
  - Alumina cantilevers, ~0.5 mm thick: robust
  - Double meander Pt heater: compensate losses by conduction
  - Includes test for resistors

Layout

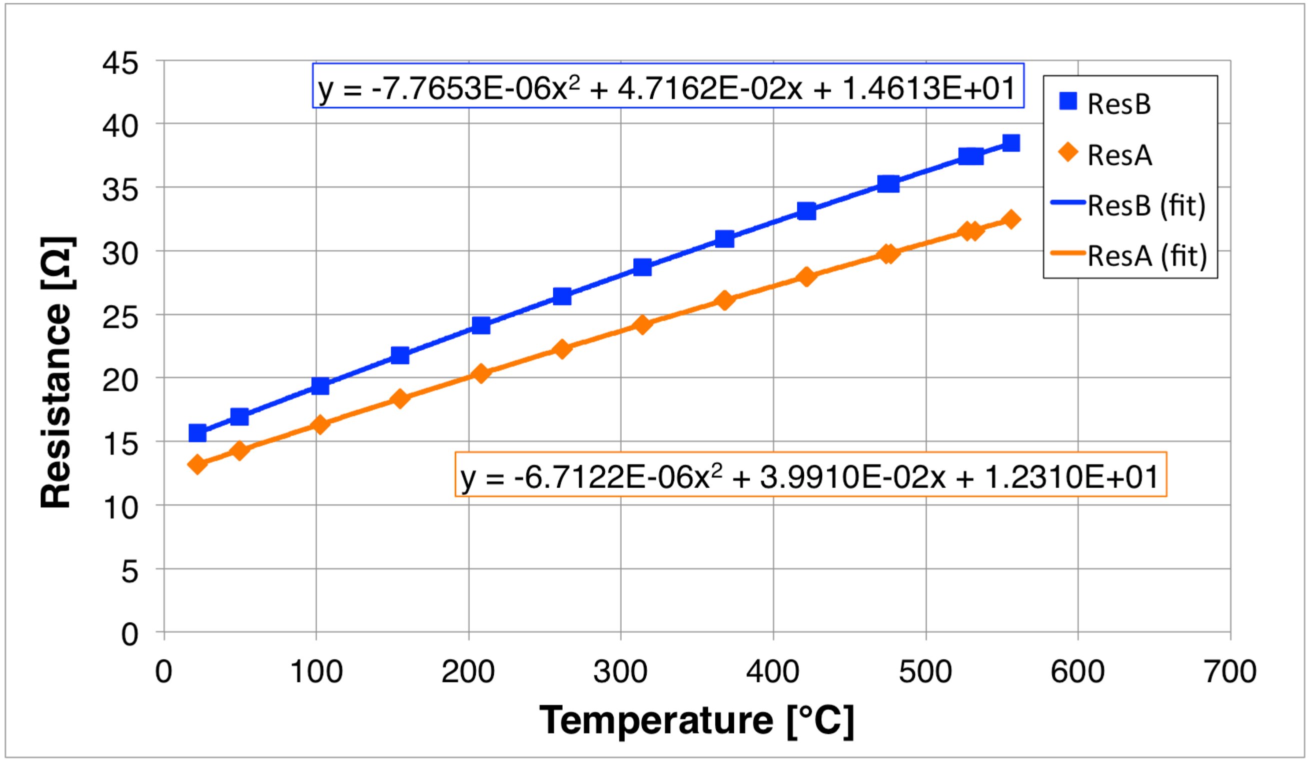


Heater calibration

- Reliable operation to ≥600°C
- Some drift above
- Acceptable to ~750°C



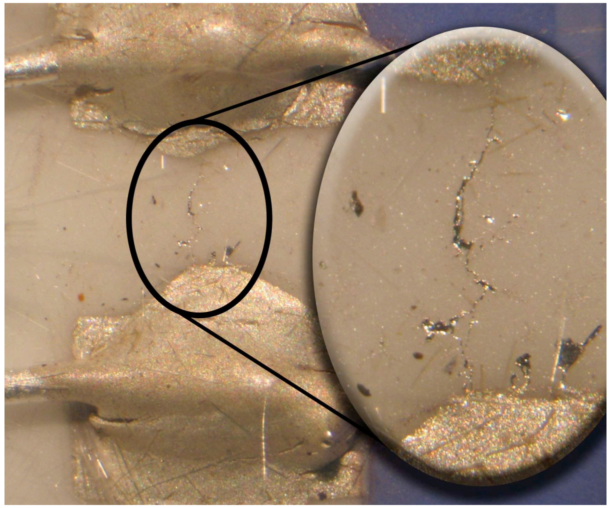
In special holder + insulation



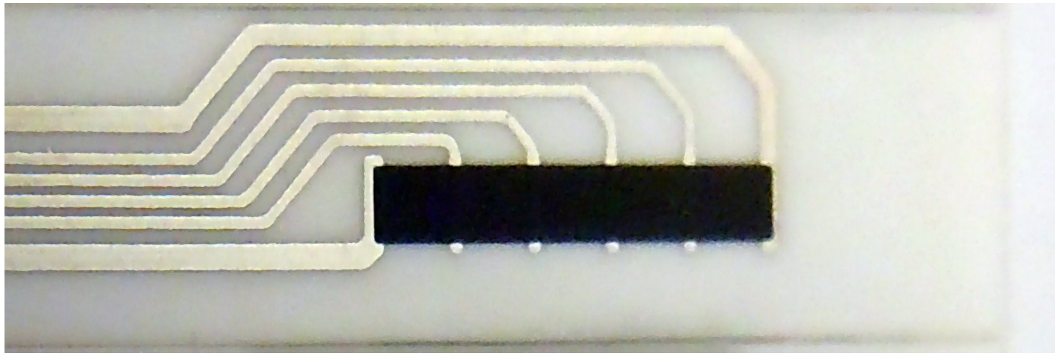
Resistor firing

- Experiments on-going
- Some issues with leakage currents at high temperatures
- Ag electromigration?
- Currently improving "shielding"

Migration of Ag



Resistor layout



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Conclusions & outlook

- **Ceramic hotplates: very versatile devices**
  - Fuel cells
  - Gas sensors
  - IR sources
  - Atomic clock components
- **Materials testing**
  - In-situ firing of resistors
  - Stability, degradation studies
  - Testing of MEMS, high-temperature chips, …
- **Outlook**
  - Long-term characterisation of reliability
  - Electromigration & other materials issues (esp. LTCC, glass)

Acknowledgments

- Swiss National Science Foundation
  - 'Sinergia' grant CRSI22-126830/1 "ONEBAT"
  - www.nonmet.mat.ethz.ch/research/onebat

- European FP7 project "CREAM"
  - AAT.2008.4.2.4-234119

- Partner labs

Glass platform – process route

1. (Bond parts of MEMS GPU)
2. Bottom: print & fire Pt heaters
3. Seal together both glass parts: also creates fluidics
4. Top: seal GPU onto carrier
5. Bottom: print & fire low-temp. Ag
6. Solder onto thick-film contact base
7. Attach fluidics connections