Ceramic cantilever hotplates for devices and testing platforms

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EPFL-LPM Laboratory for Micro-engineering for Manufacturing
Outline

1. Introduction - materials
2. Ceramic hotplates - basics
3. Fuel cells: carriers for MEMS $\mu$-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

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
The new IMT: 1 Institute on 2 Sites

Lausanne + Neuchâtel Campus

Distance: ~70 km or 45 min

http://imt.epfl.ch

1. EPFL-LPM (Lausanne)
Topics of LPM thick-film group @ EPFL

**Harsh Environments**
- Aerospace - Implantable systems
- Chemistry - Nuclear - Reactive materials
- High-temperature processes

**Load sensors**
- Force / pressure sensing
- Integration in packages
- Structuration
- Medicine / 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

- 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

1 – Processing & materials
Two main routes – typical applications

**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

- MEMS µ-SOFC reformer thermofluidic test platform
- Hotplates for high-temp. materials testing
- Dummy power components for reliability testing

- Microreactor / calorimeter
- Flow / pressure / temp. multisensor
- IR source
- Atomic clock T control
- Dummy power components for reliability testing
- Hotplates for high-temp. materials testing
Ceramic hotplates – substrate materials

**Approximate properties**

\[ T_{\text{max}} = \text{max. use temperature} \]
\[ \text{CTE} = \text{coefficient of thermal expansion} \]
\[ k = \text{thermal conductivity} \]

<table>
<thead>
<tr>
<th>Material</th>
<th>( T_{\text{max}} ) [°C]</th>
<th>CTE [ppm/K]</th>
<th>( k ) [W/m/K]</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soda-lime glass</td>
<td>520</td>
<td>8.5</td>
<td>1.0</td>
<td>Standard window glass</td>
</tr>
<tr>
<td>&quot;3.3&quot; lab glass</td>
<td>560</td>
<td>3.3</td>
<td>1.2</td>
<td>Sodium borosilicate (Pyrex, Duran, …)</td>
</tr>
<tr>
<td>Aluminoborosilicate gl.</td>
<td>730</td>
<td>3.2</td>
<td>1.2</td>
<td>Schott AF32 grade (alcali-free)</td>
</tr>
<tr>
<td>Silica glass</td>
<td>&gt;1000</td>
<td>&lt;1</td>
<td>1.3</td>
<td>Pure or almost pure ( \text{SiO}_2 )</td>
</tr>
<tr>
<td>LTCC (DP 951)</td>
<td>580</td>
<td>~6</td>
<td>~3</td>
<td>Very common LTCC material</td>
</tr>
<tr>
<td>LTCC (Her CT800)</td>
<td>&gt;750</td>
<td>~6</td>
<td>~3-4</td>
<td>More crystallising LTCCs</td>
</tr>
<tr>
<td>LTCC (CT GC)</td>
<td>&gt;750</td>
<td>~6</td>
<td>~3-4</td>
<td></td>
</tr>
<tr>
<td>Alumina (( \text{Al}_2\text{O}_3 ) 96%)</td>
<td>&gt;1000</td>
<td>~7</td>
<td>~20</td>
<td>Standard ceramic substrate</td>
</tr>
<tr>
<td>Zirconia (stabilised)</td>
<td>&gt;1000</td>
<td>~11</td>
<td>~3</td>
<td>Strength, ionic conductor</td>
</tr>
</tbody>
</table>

\( T_{\text{max}} \) = max. use temperature

- CTE = coefficient of thermal expansion
- \( k \) = thermal conductivity

Approximate properties

1 – Processing & materials

36th IMAPS-CPMT Poland, Kołobrzeg, 26-29.9.2012
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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
- $L \sim 50 \text{ mm}$
- $b = 7 \text{ mm}$
- $h = 250...500 \mu \text{m}$

Gas multisensor
- $L \sim 2-3 \text{ mm}$
- $b = 7 \text{ mm}$
- $h = 100...200 \mu \text{m}$

$\mu$-SOFC platform
- $L \sim 7 \text{ mm}$
- $b \sim 2 \text{ mm} \text{ (total)}$
- $H \sim 1 \text{ mm} \text{ (fluidics)}$
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### Class

<table>
<thead>
<tr>
<th>Material</th>
<th>$k$ [W/m/K]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glasses</td>
<td>0.5…1.5</td>
</tr>
<tr>
<td>LTCC</td>
<td>3…5</td>
</tr>
<tr>
<td>Zirconia (stabilised)</td>
<td>~3</td>
</tr>
<tr>
<td>Alumina ($\text{Al}_2\text{O}_3$ 96%)</td>
<td>15…25</td>
</tr>
</tbody>
</table>

**Ranking (heat conduction)**

- Best: LTCC (structuration)
- Good: glass & $\text{ZrO}_2$
- Less optimal: $\text{Al}_2\text{O}_3$
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**Atomic clock T control**

\[ T_{\text{cold}} \sim 25^\circ \text{C} \]
\[ T_{\text{hot}} \sim 80^\circ \text{C} \]
\[ W_{\text{cond}} \sim 1'000 \text{ K/W} \]
\[ W_{\text{observed}} \sim 80 \text{ K/W (in free air)} \]

**µ-SOFC platform**

\[ T_{\text{cold}} \sim 50^\circ \text{C} \]
\[ T_{\text{hot}} \sim 550^\circ \text{C} \]
\[ W_{\text{cond}} \sim 400 \text{ K/W} \]
\[ W_{\text{observed}} < 100 \text{ K/W (in insulated oven)} \]

**Countermeasures**

- Insulation
- Radiation shields
- Vacuum
Thermomechanical decoupling

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Suspended by bond wires (common in classical gas sensors)

Good properties
Cumbersome process…
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Thermal stresses!
Only OK for small $\Delta T$
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Cantilevers

Structured hotplate

2005 Kita

2 - Hotplate basics
Thermomechanical decoupling

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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)*
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

Power requirements

Portable PC: ~50 W

Media player: ~1…5 W

Camcorder: ~10 W

3 – Carriers for μ-SOFC elements

36th IMAPS-CPMT Poland, Kołobrzeg, 26-29.9.2012
**µ-SOFC system principle**

- Integration into a single, compact module

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**µ-SOFC module**

- Fuel tank
- Control
- Battery
- Heat
- Exhaust
- Power output

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3 – Carriers for µ-SOFC elements
μ-SOFC system issues

- Gas reforming
- Electrochemical conversion
- Post-combustion

**μ-SOFC module principle**

**µ-SOFC module hot module flows & interactions**

- **Internal heat exchanges & losses to ambient**

**Electrical power**

**Air** → **Fuel** → **Reformer** → **Fuel cell** → **Post-combustor** → **Exhaust**
LTCC $\mu$-SOFC module concept

- Simple "stick" structure
- Facile thermal & mechanical decoupling
- External (electrical & fluidic) connections at low temperature
- Integration of fluidics & possibly gas processing
LTCC $\mu$-SOFC module concept

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LTCC $\mu$-SOFC module

PCU = post-combustor
GPU = reformer
LTCC $\mu$-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
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

Top side
Fluidic connections & GPU seal

Bottom (Si) side
Heating/sensing tracks

MEMS Gas processing unit (GPU)
Main Pt heater
Guard Pt heater

3 – Carriers for µ-SOFC elements
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

![Diagram showing temperature and power consumption over time]

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)
- \( C_xH_y + O_2 \rightarrow H_2, CO \) 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)
- \( \text{C}_x\text{H}_y + \text{O}_2 \rightarrow \text{H}_2, \text{CO} \) over catalyst in GPU

1. Disappearance of propane / butane gas
2. Syngas yield = \((\text{H}_2 + \text{CO}) / \text{ideal}\)

![Graphs showing the relationship between temperature and syngas yield for Butane and Propane](image)

- 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)

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2007 Moos (IMAPS-PL)

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Gas side

Gas electrode

ZrO$_2$ electrolyte

Air electrode + channel

Heater

3 – Carriers for µ-SOFC elements
Related: potentiometric oxygen sensor

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

Reference = air channel

Reference = buffer

2007 Moos (IMAPS-PL)

2012 Kobierowska & al. (IMAPS-PL)
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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

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**Basic principle**
- Radiant heater
- Hot zone
- Cold outer frame

**Radiant heater**

**Hotplate for gas sensor**

2005 Kita
Compact hotplates – process issues

- 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

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- Fully cut out, may break during processing
- 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](image-url)
Heater calibration

- Reliable operation to $\geq 600^\circ C$
- Some drift above
- Acceptable to $\sim 750^\circ C$

In special holder + insulation

\[
\begin{align*}
\text{Temperature [^\circ C]} & \quad \text{Resistance [\Omega]} \\
0 & \quad 0 \\
100 & \quad 10 \\
200 & \quad 15 \\
300 & \quad 20 \\
400 & \quad 25 \\
500 & \quad 30 \\
600 & \quad 35 \\
700 & \quad 40 \\
\end{align*}
\]

\[
y = -7.7653E-06x^2 + 4.7162E-02x + 1.4613E+01
\]

\[
y = -6.7122E-06x^2 + 3.9910E-02x + 1.2310E+01
\]
Resistor firing

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

Resistor layout

Migration of Ag
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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