

Structuration of Micro-fluidic Devices Based on Low Temperature Co-fired Ceramic (LTCC) Technology

H. Birol, T. Maeder, C. Jacq, S. Straessler & P. Ryser

**Swiss Federal Institute of Technology, Lausanne - EPFL
Laboratory for Production of Microtechnologies - LPM
Thick-film Group**

lpmwww.epfl.ch

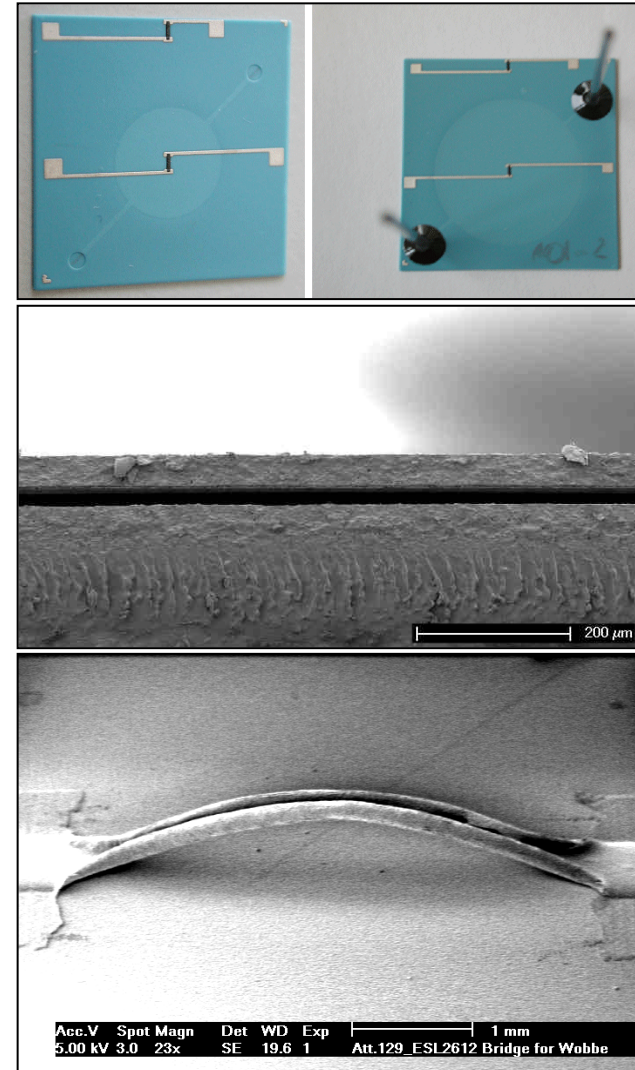


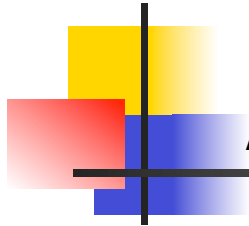
PURPOSE OF THE PRESENTATION

➔ Preparation of **graphite-based sacrificial layer** for microstructuring of LTCC

➔ To demonstrate the fabricated structures; **membranes**

➔ To explain the **processing conditions** and their effects on final device properties



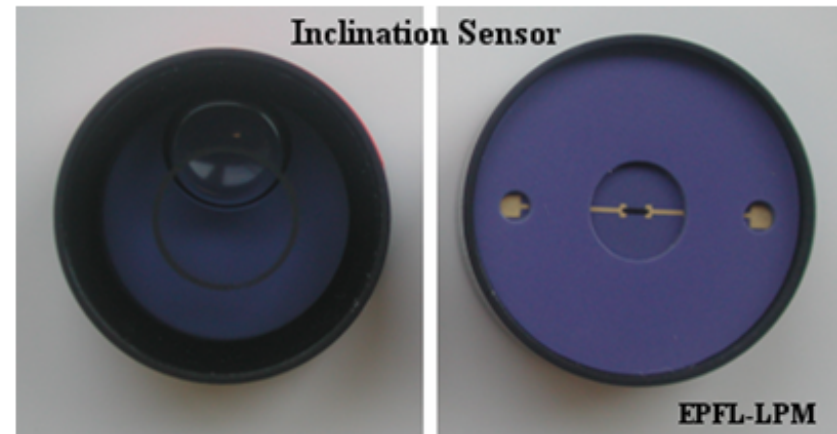
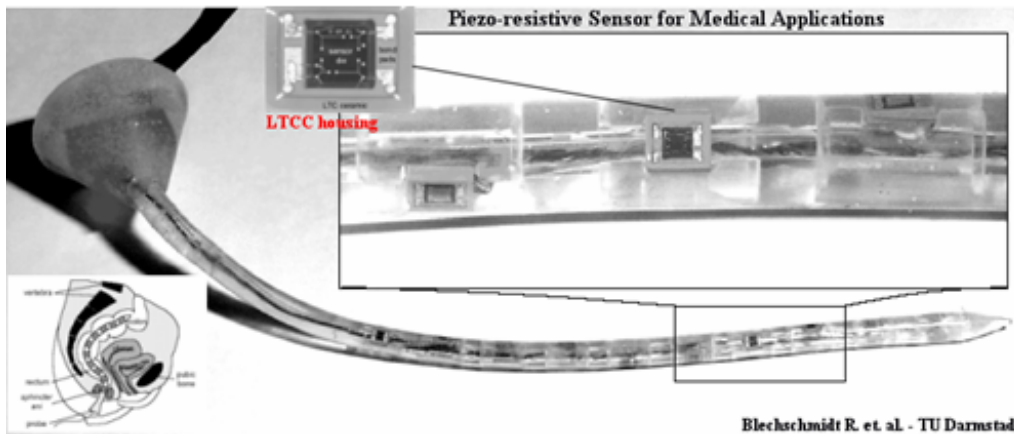
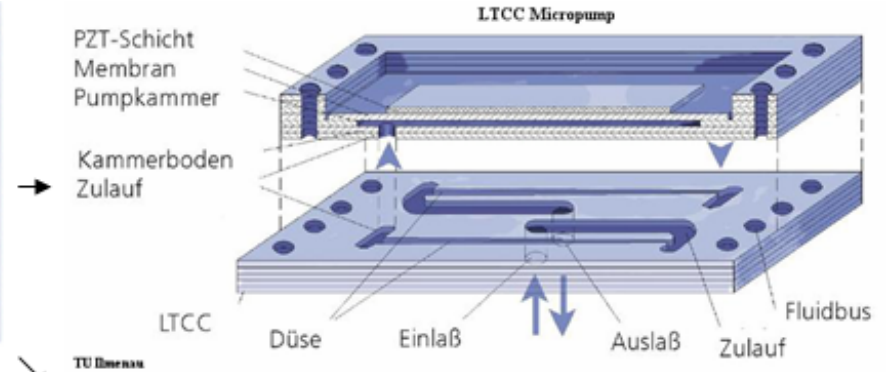
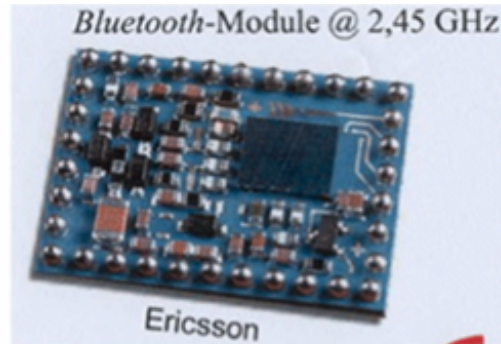


AN OVERVIEW

Application areas of LTCC technology have diversified



Source: Primark/Binghamton University



→ What are the challenges and solutions for 3-D structuration?



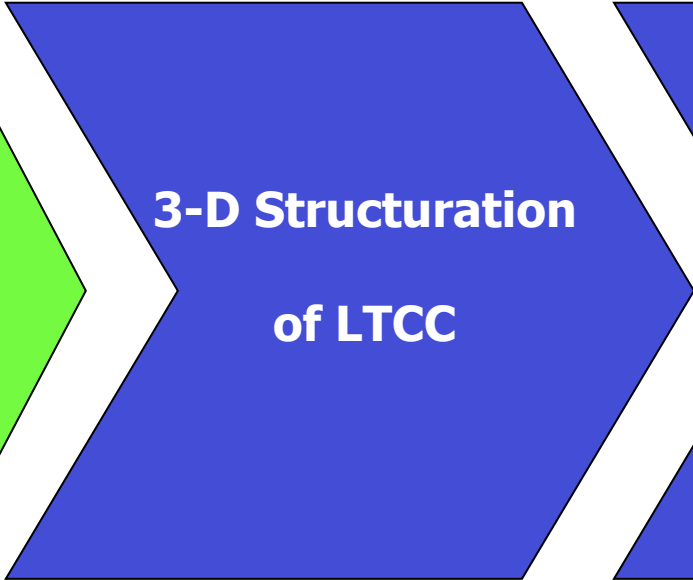


OUTLINE OF THIS PRESENTATION



LTCC Technology for Micro-fluidic Sensor Applications

- Introduction
- Advantages
- Challenges



3-D Structuration of LTCC

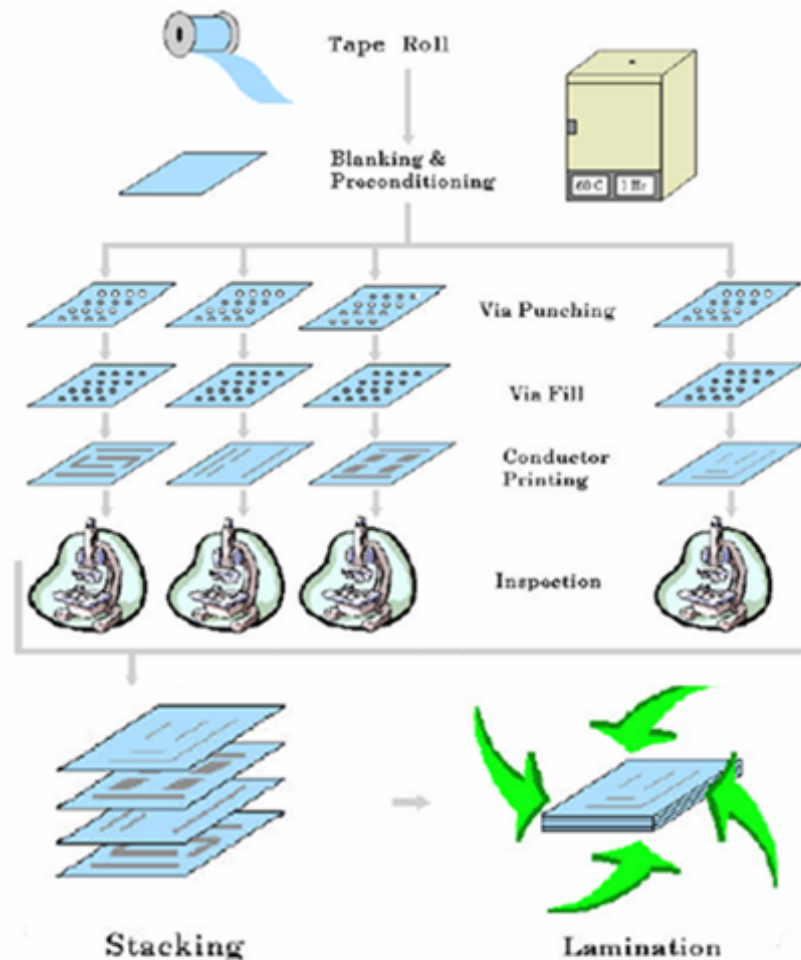
- Carbon-sacrificial
paste preparation
- Fabricated Structures



Discussions

- Material Properties
- Effects of processing
conditions on structure
- Conclusions and next steps

INTRODUCTION: LTCC MATERIALS SYSTEM



Ceramic / glass mixture blended in organic vehicle & cast into thick-film sheets (LTCC tapes: 30-350 um)

Layers processed by

- cutting,
- screen-printing pastes

→ **CO-FIRING & COMPATIBILITY** with thick-film technology

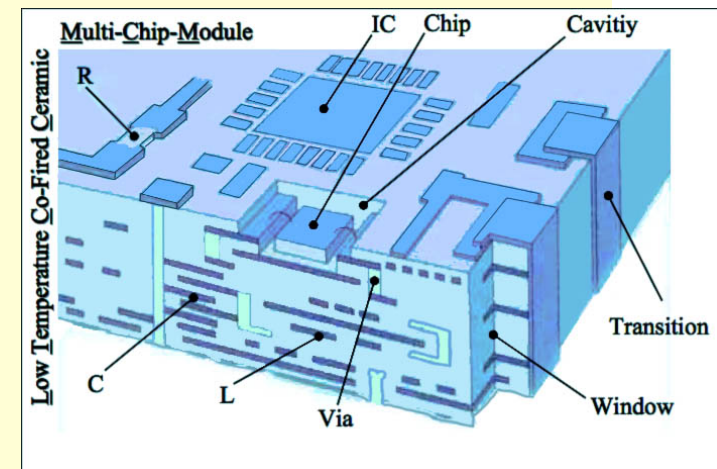
Lamination up to **80 layers**

Firing **< 900°C**

by Heraeus

ADVANTAGES OF LTCC FOR MICRO-FLUIDIC APPLICATIONS

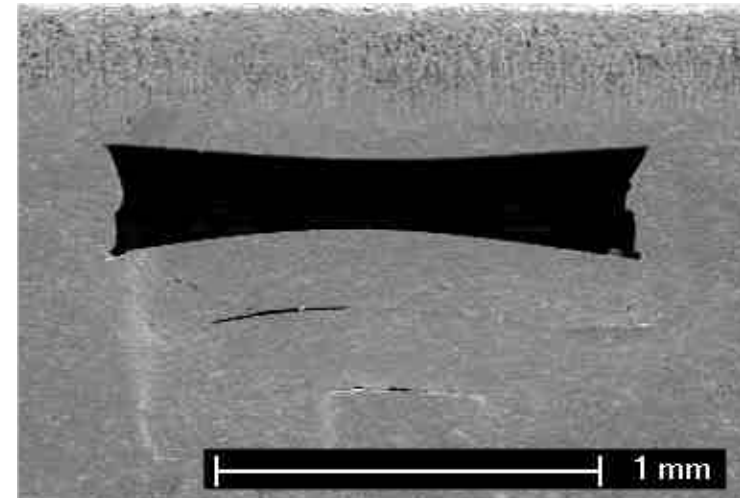
- Excellent chemical / thermal stability
- Ease of machinability of tapes
- Cost effective
- High density packaging
- Hermeticity of the structures
- Mechanical and electrical functions in one system



Sagging

Unsupported cavity is deformed during:

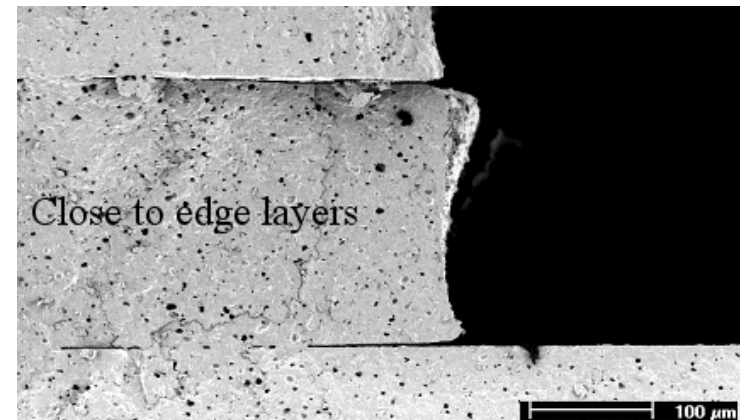
- Lamination (lamination stress)
- Sintering (over T_g)



Delamination / Disintegration

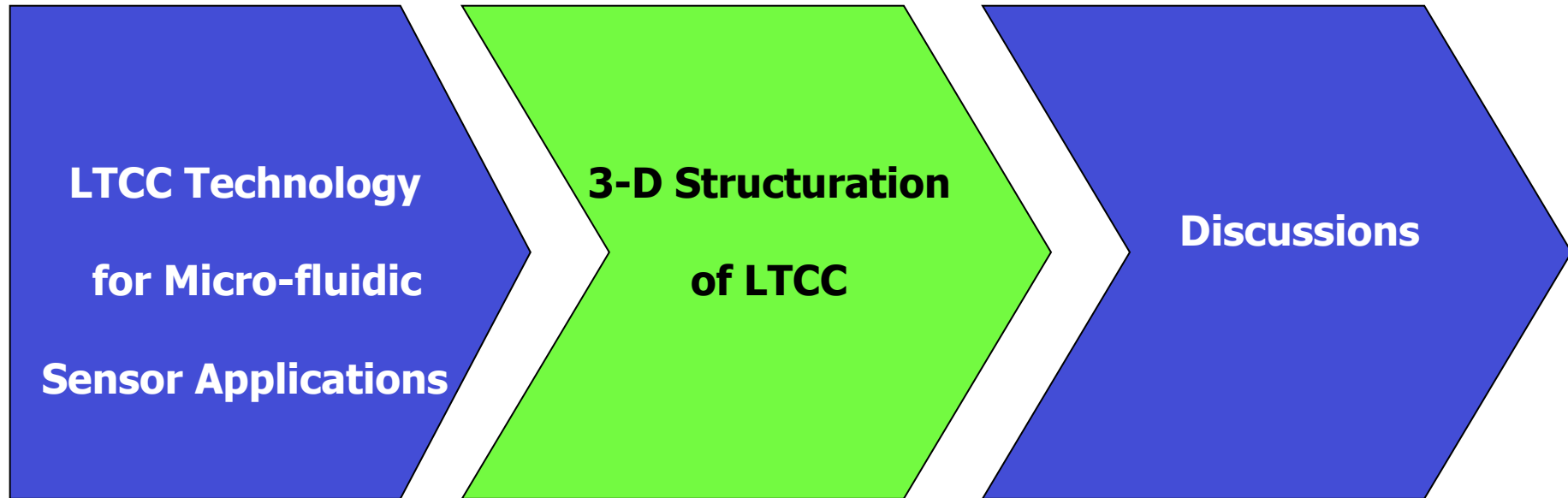
Occurs due to:

- Poor lamination
- Geometrical constraints





OUTLINE OF THIS PRESENTATION



→ Introduction

→ Advantages

→ Challenges

→ Carbon-sacrificial
paste preparation

→ Fabricated Structures

→ Material Properties

→ Effects of processing
conditions on structure

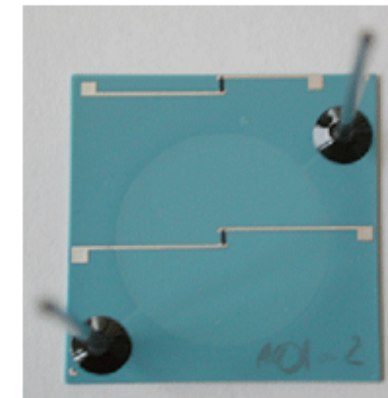
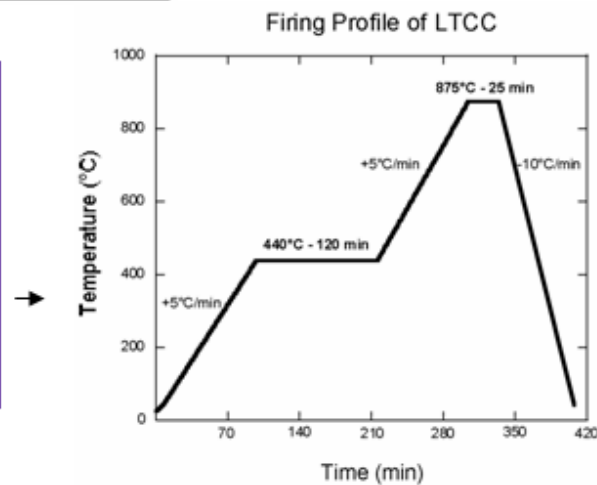
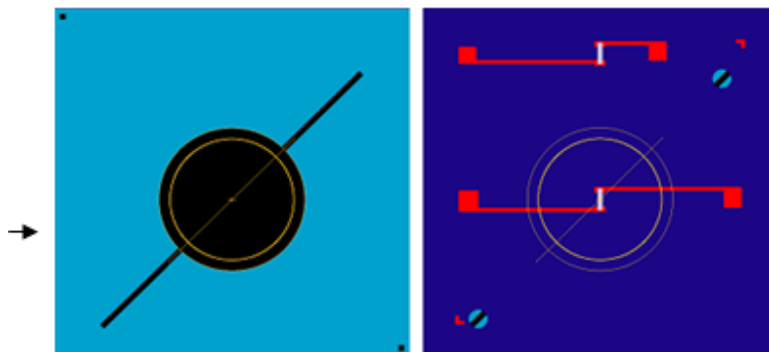
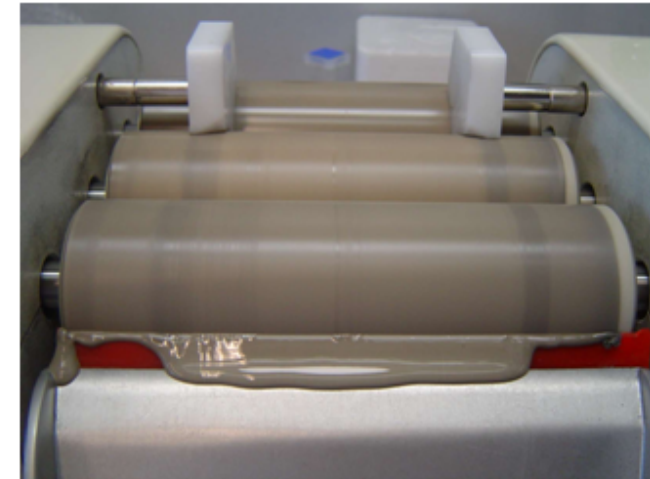
→ Conclusions and next steps

CARBON SACRIFICIAL PASTE PREPARATION

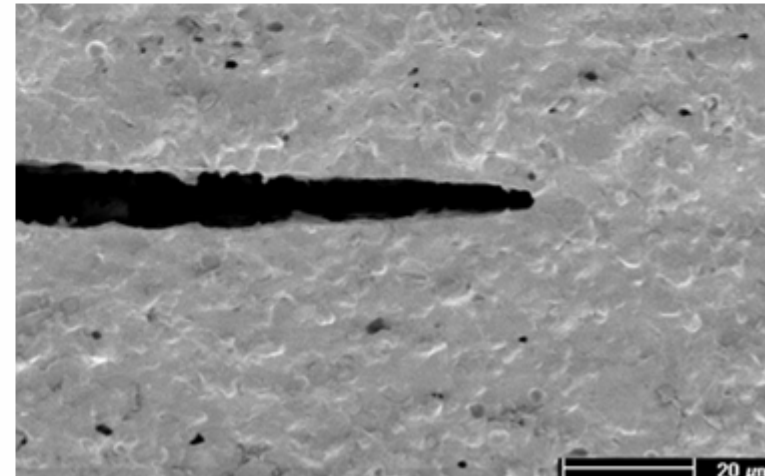
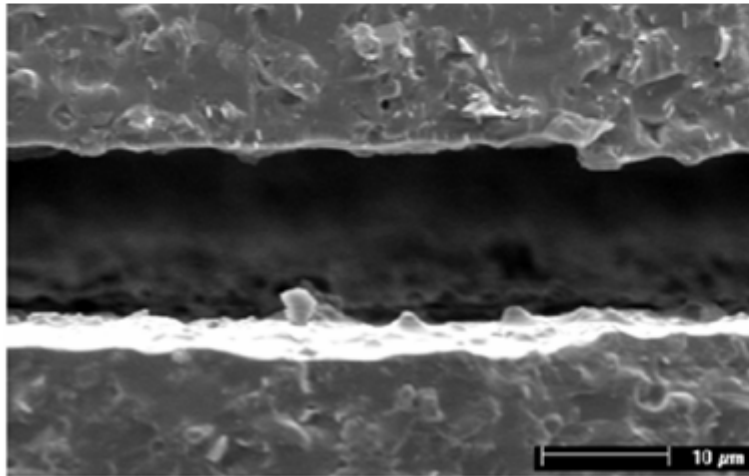
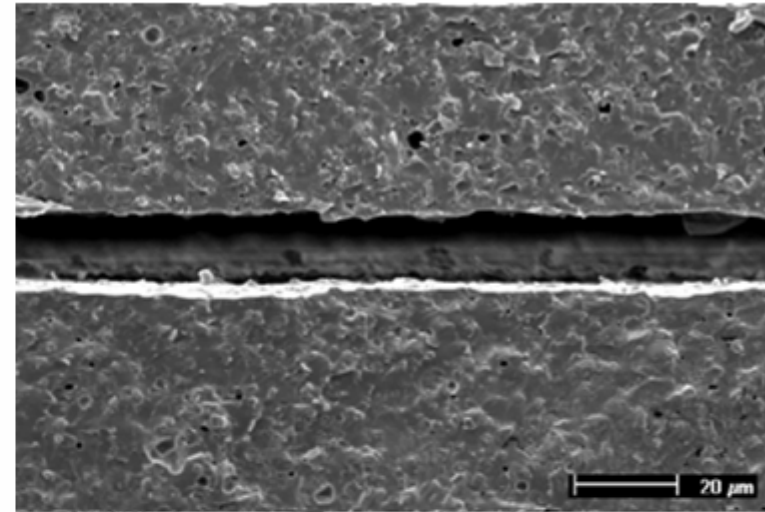
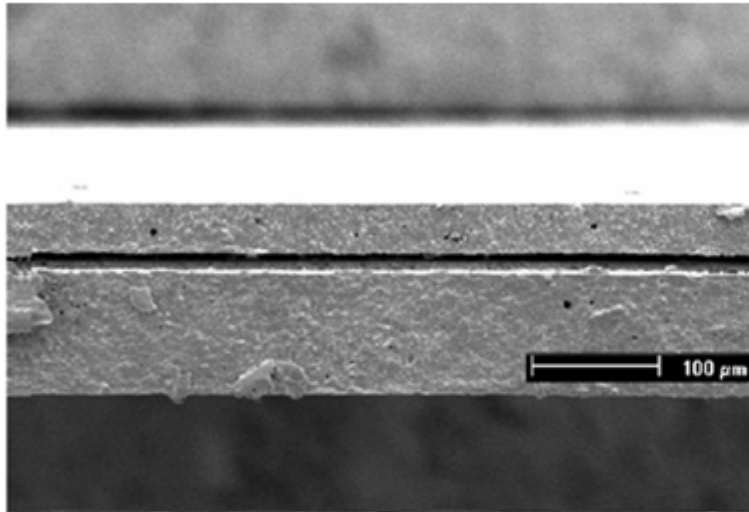
Product	Function	Specification	Supplier
Graphite	Sacrificial	$d_{50} : 1-2\mu\text{m}$ ★	Aldrich, 28,286-3
		$d_{50} : 11\mu\text{m}$	TIMCAL, Timrex-KS25
		$d_{50} : 15.3\mu\text{m}$ ★	TIMCAL, Timrex-KS5-25
Ethyl cellulose	Binder	control of rheology	Aldrich, 43,383-7
Terpineol	Solvent	slurry viscosity	Fluka, 86480
Acetyl acetone	Dispersant	dispersing additive	Sigma-Aldrich, P775-4

Graphite
26 wt%

Organics
74 wt%

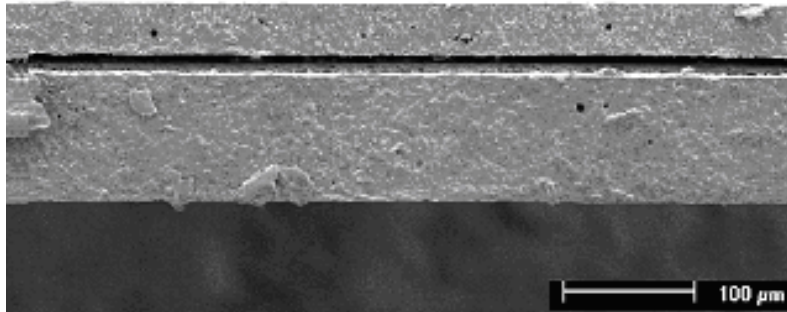


FABRICATED STRUCTURES

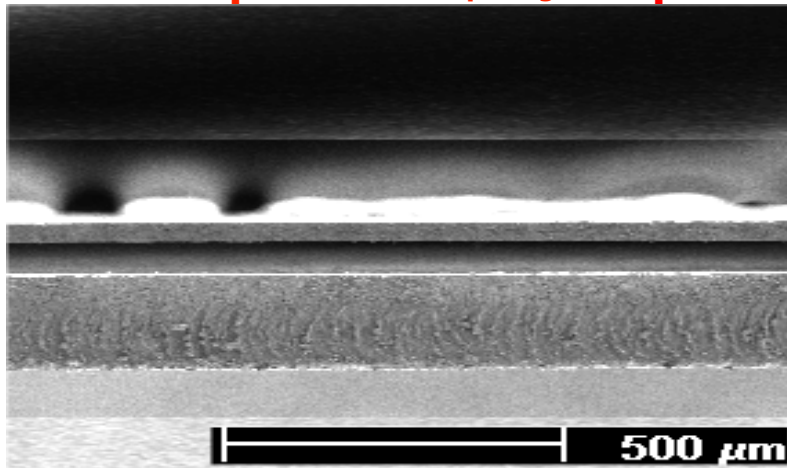


FABRICATED STRUCTURES

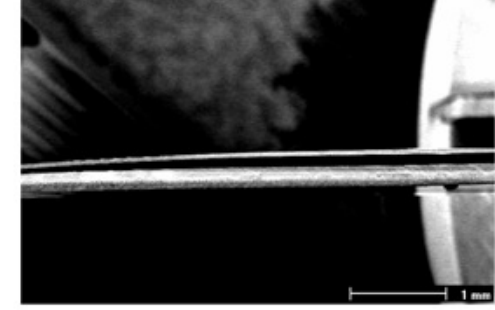
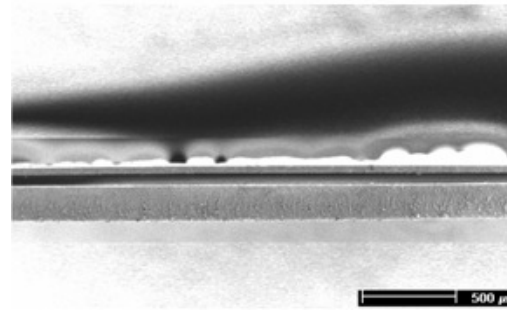
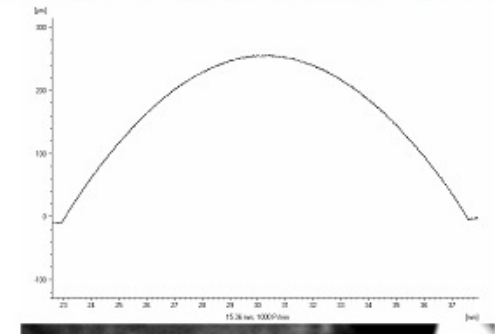
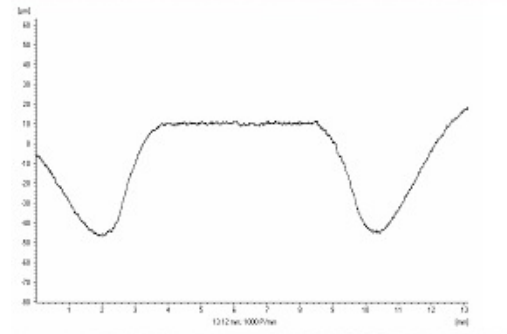
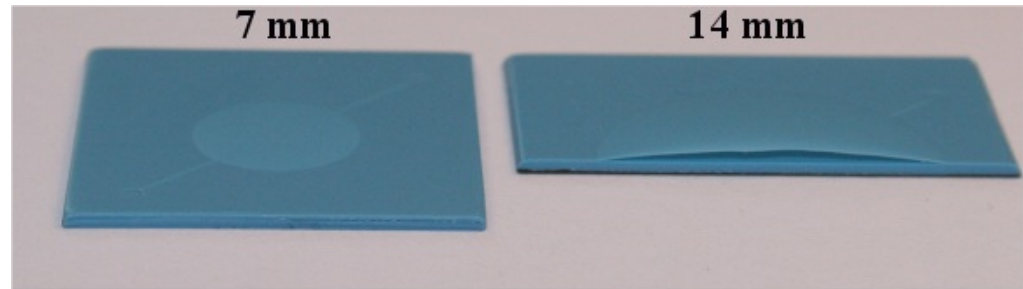
$d_{50} \sim 2\mu\text{m}$ \rightarrow $t_{\text{spacing}} \sim 13\mu\text{m}$

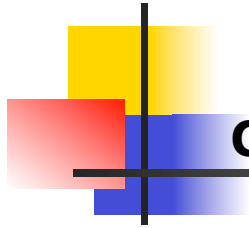


$d_{50} \sim 15\mu\text{m}$ \rightarrow $t_{\text{spacing}} \sim 60\mu\text{m}$

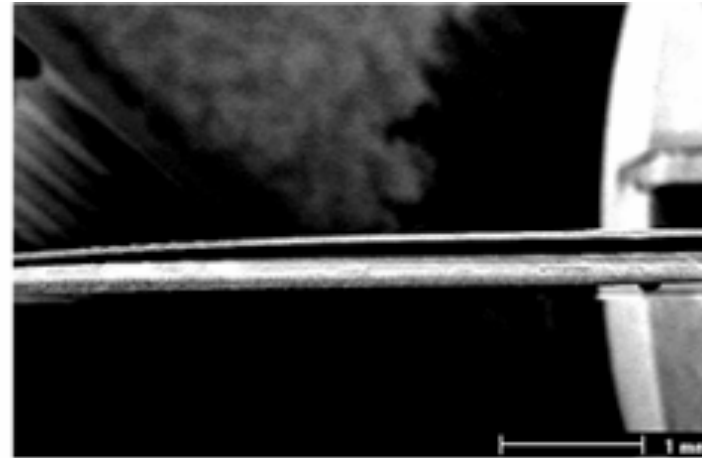
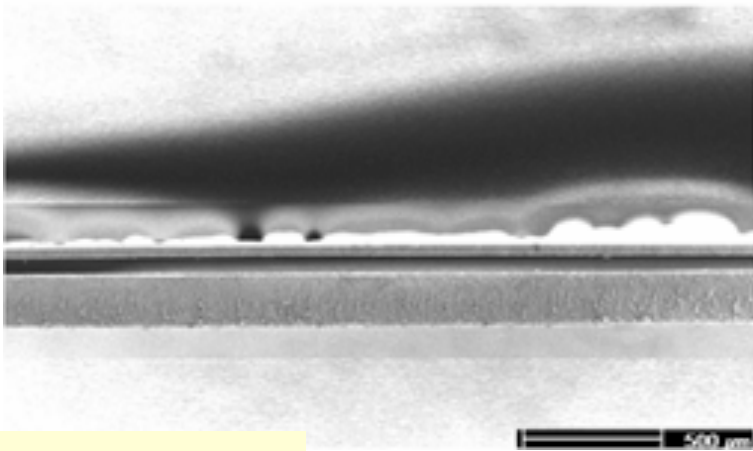


$d_{50} \sim 15\mu\text{m} \rightarrow$ Swelling

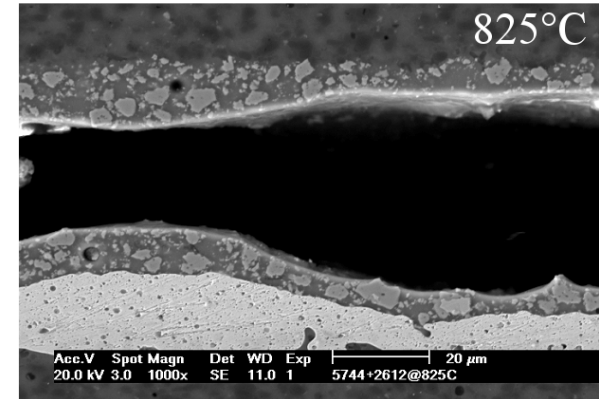
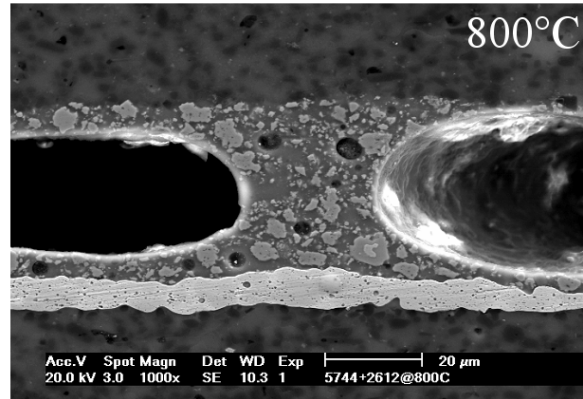
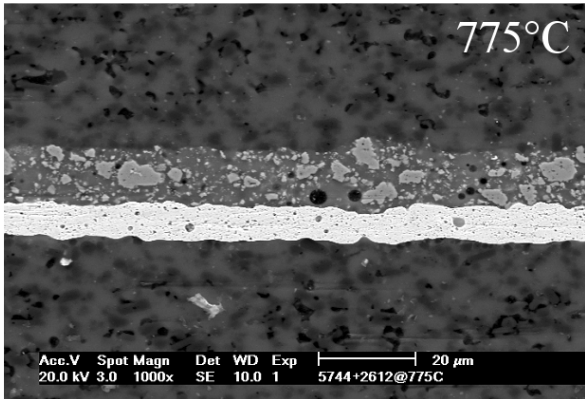




CORRELATION



Membranes



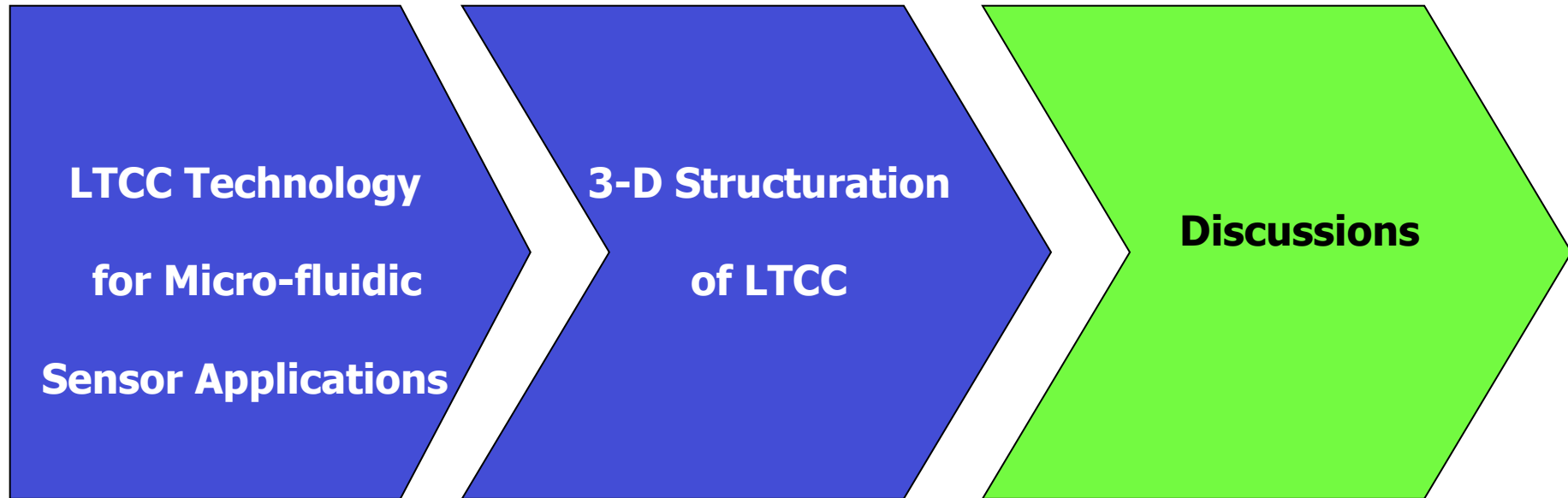
Buried TFR

→ What is the origin of swelling (at 750-800°C) ?





OUTLINE OF THIS PRESENTATION



→ Introduction

→ Advantages

→ Challenges

→ Carbon-sacrificial
paste preparation

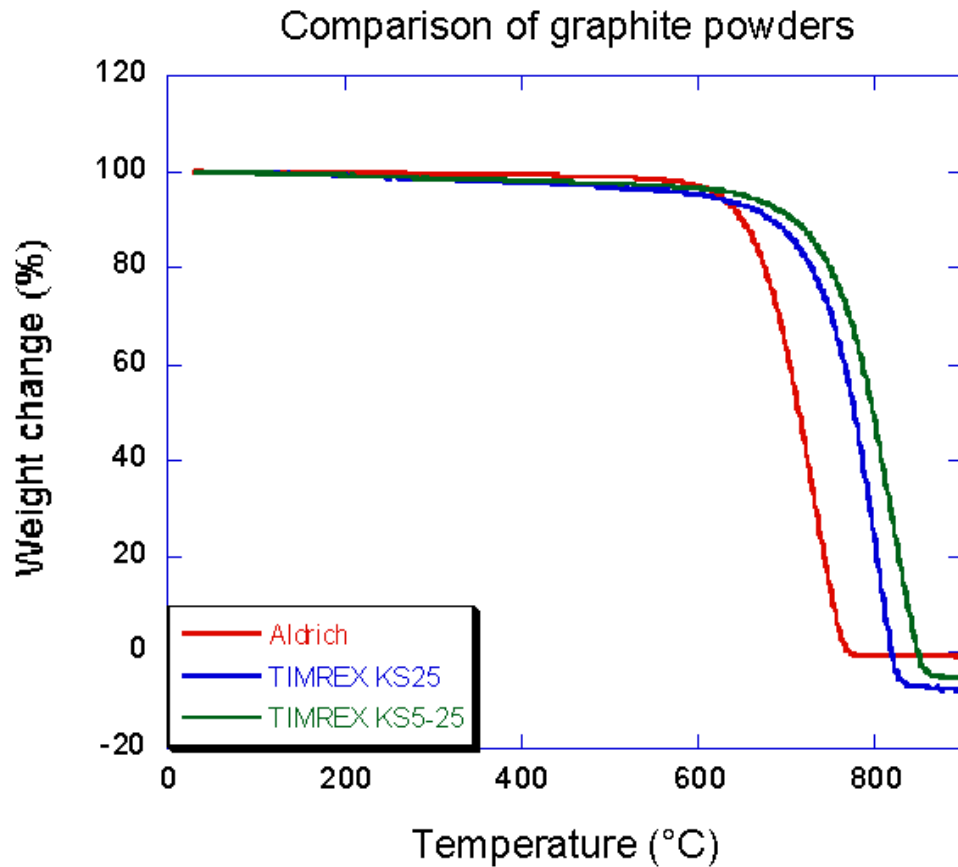
→ Fabricated Structures

→ Material Properties

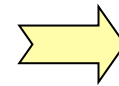
→ Effects of processing
conditions on structure

→ Conclusions and next steps

CHARACTERISTICS OF GRAPHITE and LTCC



→ Heating rate of 10°C/min in air!

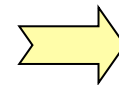


Burnout temperature of graphite;

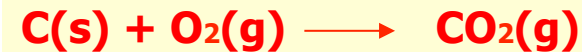
d₅₀ ~ 2µm → 770°C

d₅₀ ~ 15µm → 860°C

... 1



Ideal graphite burnout reaction;



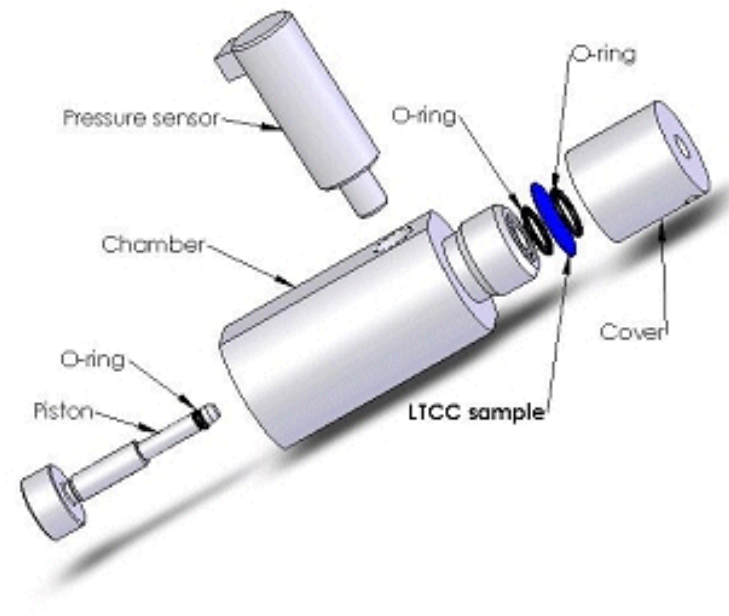
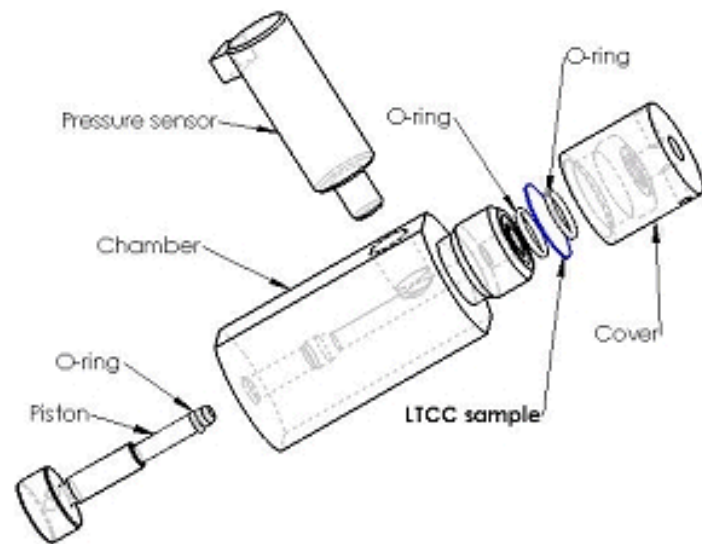
What is the final temperature for full-degassing of burnt products?

OR

What is the temperature for elimination of LTCC open-porosity?

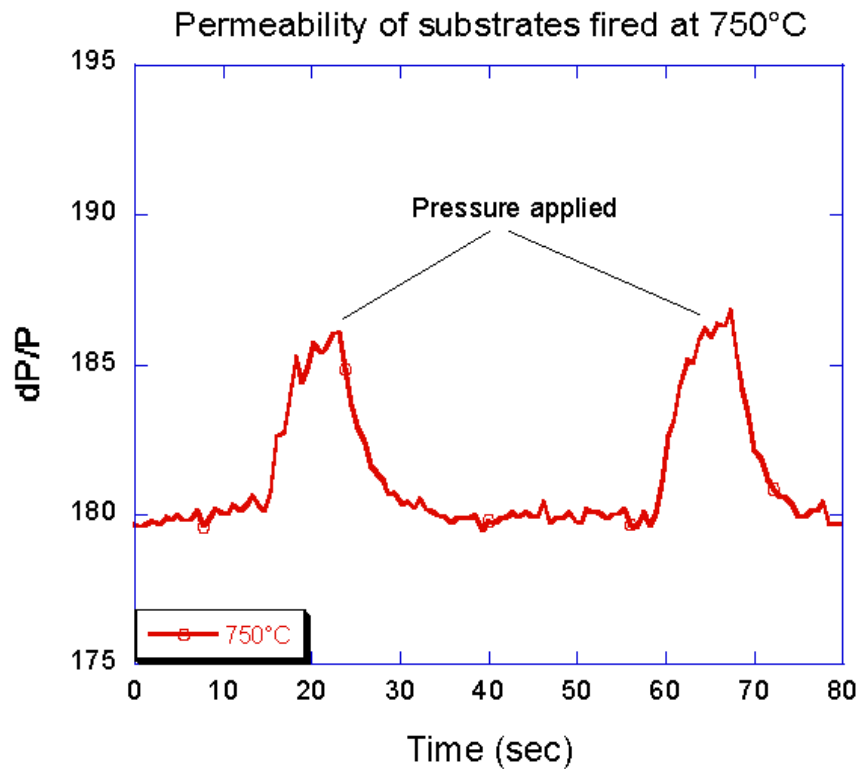
DETERMINATION of LTCC OPEN-POROSITY ELEMINATION TEMPERATURE

Closed-chamber for determination of open-porosity elimination temperature

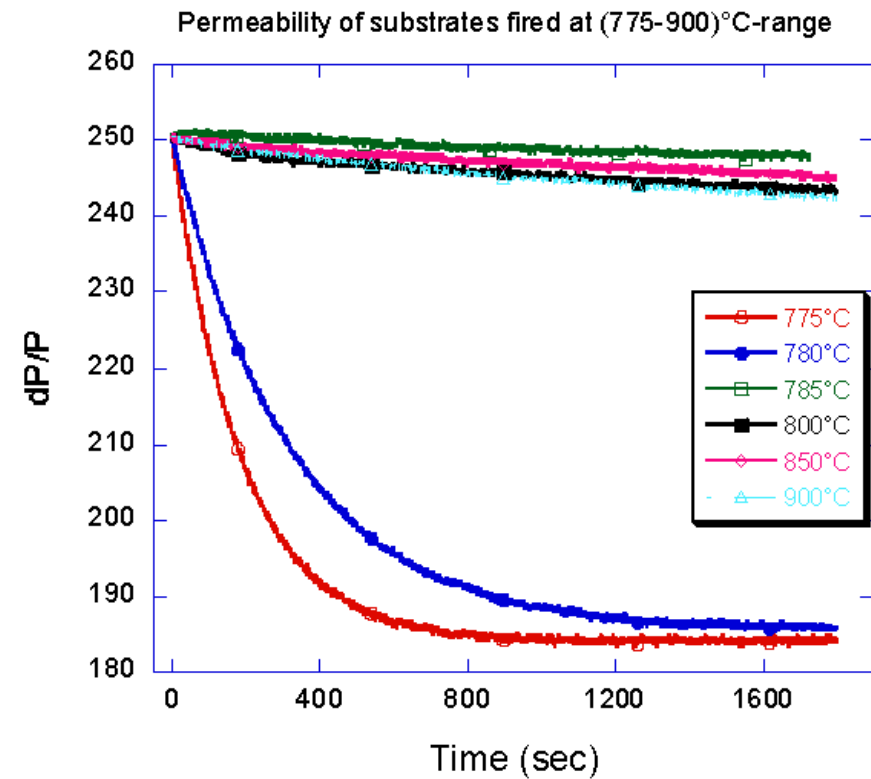


Air leakage through LTCC is detected via pressure-relaxation time

DETERMINATION of LTCC OPEN-POROSITY ELEMINATION TEMPERATURE



Permeable substrate at 750°C

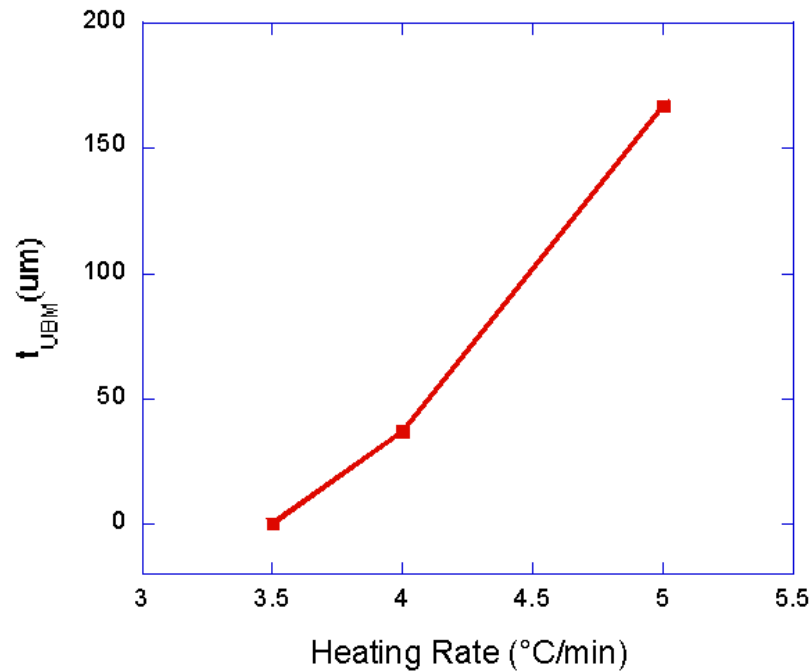


Open-porosity eliminated at ~ 785°C

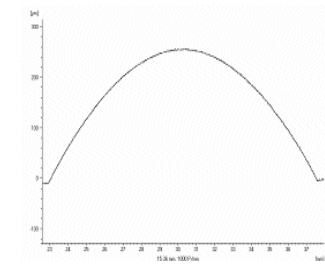
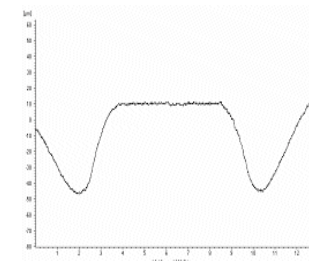
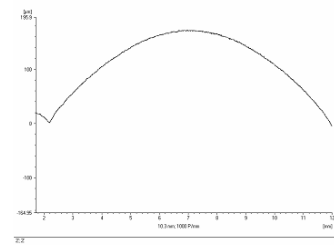
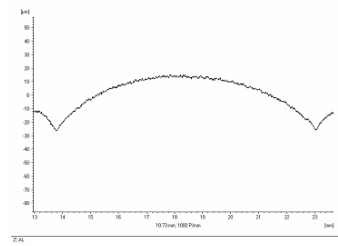
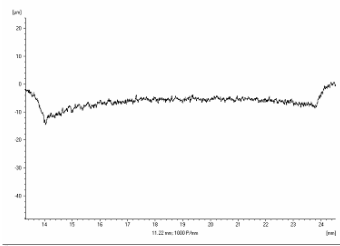
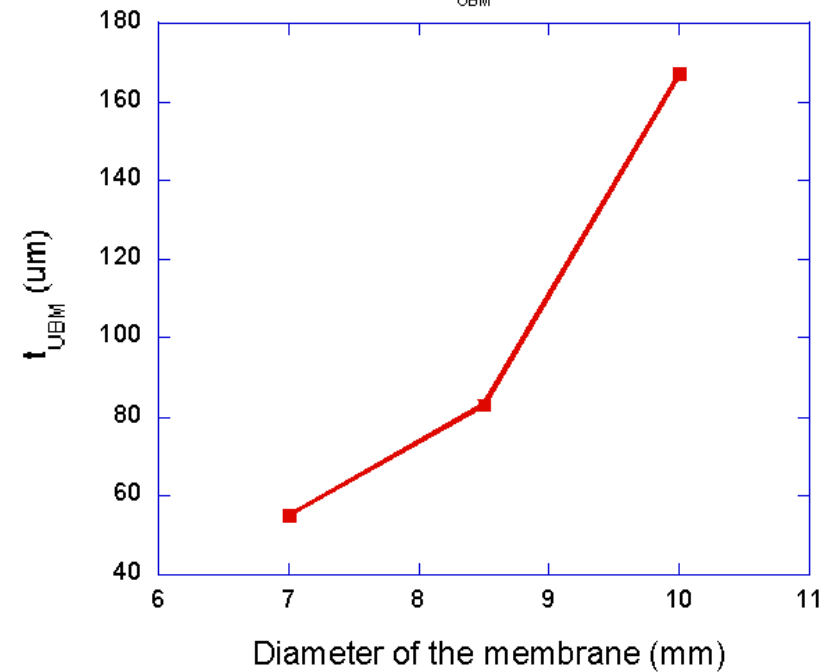
...2

EFFECT OF PROCESSING CONDITIONS ON STRUCTURE

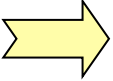
Effect of heating rate on t_{UBM} (diameter=10mm)



Effect of diameter on t_{UBM} (Heating rate=5°C/min)



ORIGIN OF MEMBRANE CHARACTERISTICS



Graphite powder	T _{burnout} (°C) [*]	T _{LTCC-open-pore elimination} (°C) ⁺	Unburned (%)
Fine (d ₅₀ ~2µm)	770	785	0
Coarse (d ₅₀ ~15µm)	865		less than 55

* : Heating in air at 10°C/min rate

+ : Heating in air at 5°C/min rate

The **spacing** and **membrane flatness** is determined by the competition between the kinetics of

- **the graphite burnout**
- **LTCC sintering**

- The chemistry,
- Particle size (d₅₀),
- Heating rate,
- Membrane diameter,
- Thickness

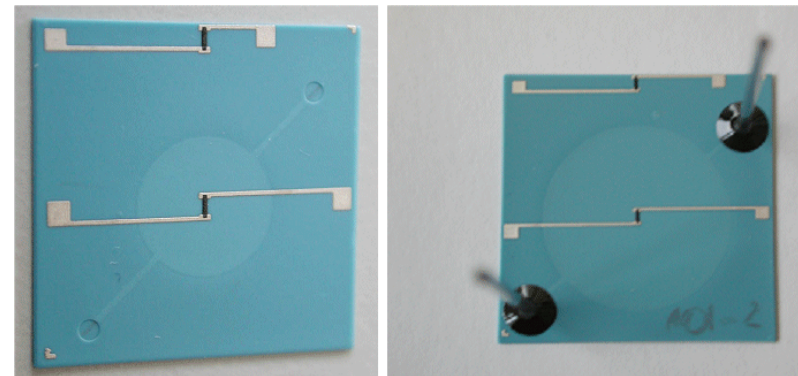
Closure of LTCC porosity leads to **depletion of oxygen** to burn the graphite, which shifts the CO/CO₂ equilibrium according to



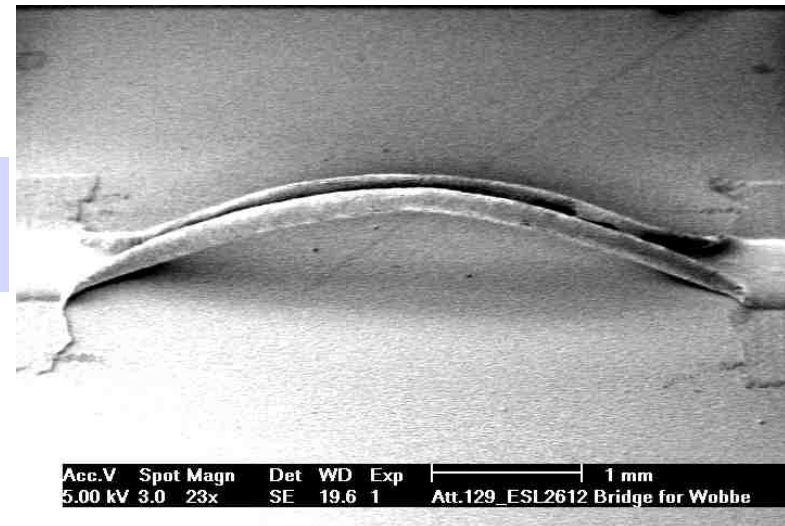
CONCLUSIONS

- Carbon sacrificial pastes effectively used for fabrication of membranes/channels
- Membranes produced with
 - **thickness**: of 40 μ m
 - **diameter**: 7,10, 15, 18mm
 - **spacing**: (10-100 μ m)
- Final membrane features dependent on **graphite powder** and **LTCC properties**

Desired structures can be fabricated selecting the **right processing parameters**



14mm-diameter-membrane with PTC resistors



PTC free-hanging bridge on LTCC



NEXT STEPS

- Investigation of different paste compositions (permanent sacrificials)
- Improving the lamination technique
- Application of non-destructive testing methods e.g. ultrasonic microscope, for examining the entire structure
- Measurements with the prototypes