Preparation and Application of "Minerals-based Sacrificial Pastes" for Fabrication of LTCC Structures

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Purpose of this presentation

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- Fugitive-phases for fabrication of LTCC:
  Overview & comparison
- Minerals-based sacrificial pastes:
  Motivation & introduction
- Preliminary results:
  Materials used & fabricated structures

An overview of LTCC technology

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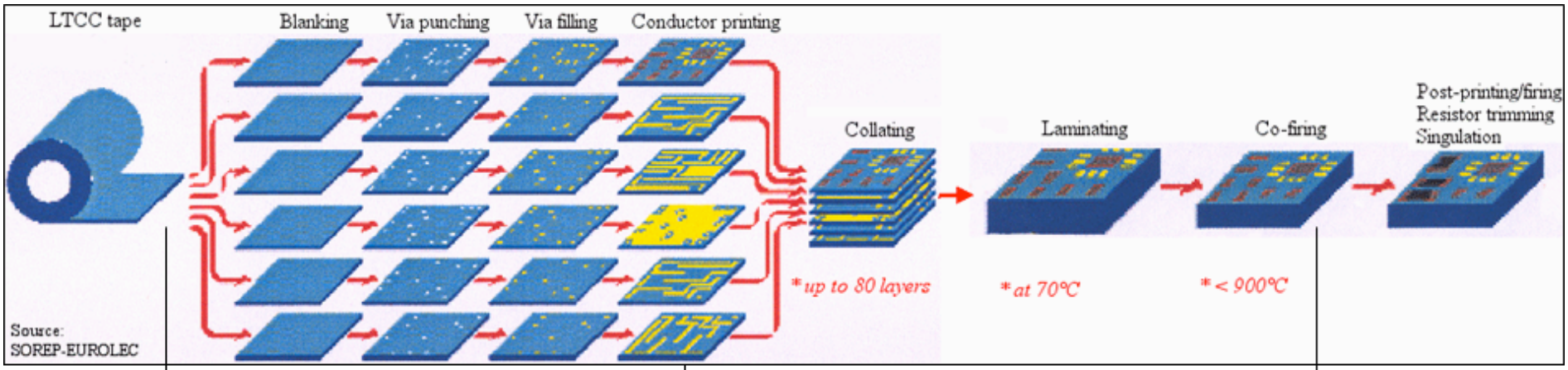
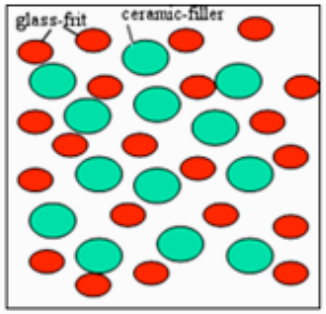
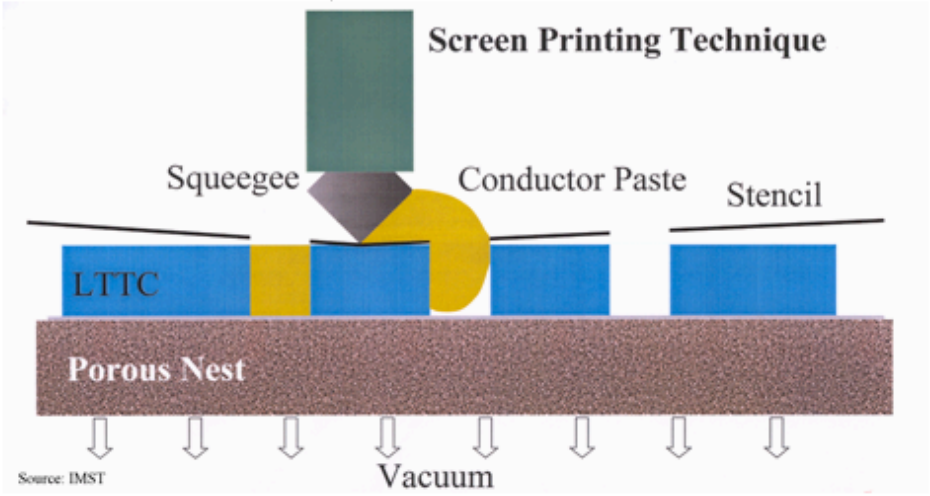
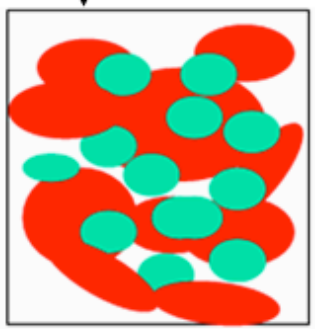




Glass-frit: low firing T
Filler: dimensional stability

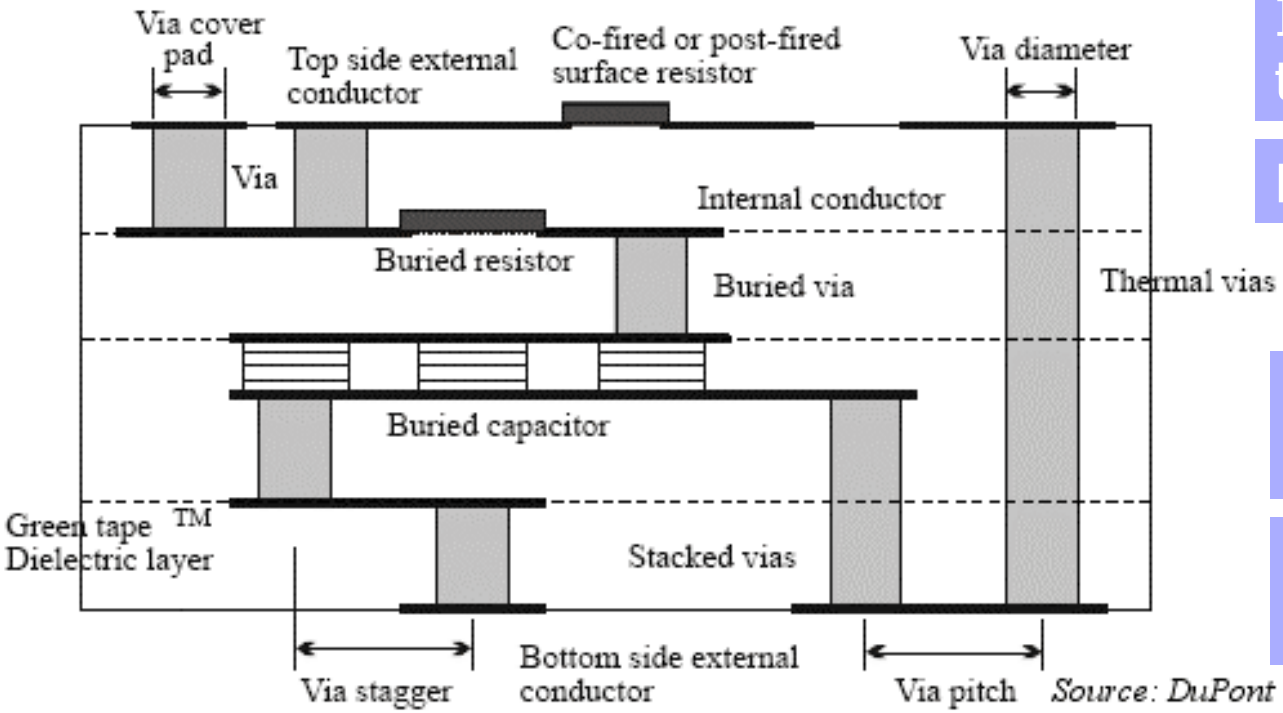
... blended in organic-vehicle & cast on mylar sheets





Glass wetting and ceramic rearrangement occur

LTCC for sensors & fluidics



- Machinability of tapes
- Integration of thick-film technology
- High density packaging
- Chemical / thermal stability
- Mechanical and electrical functions in one system
- Cost effective

Outline of this presentation

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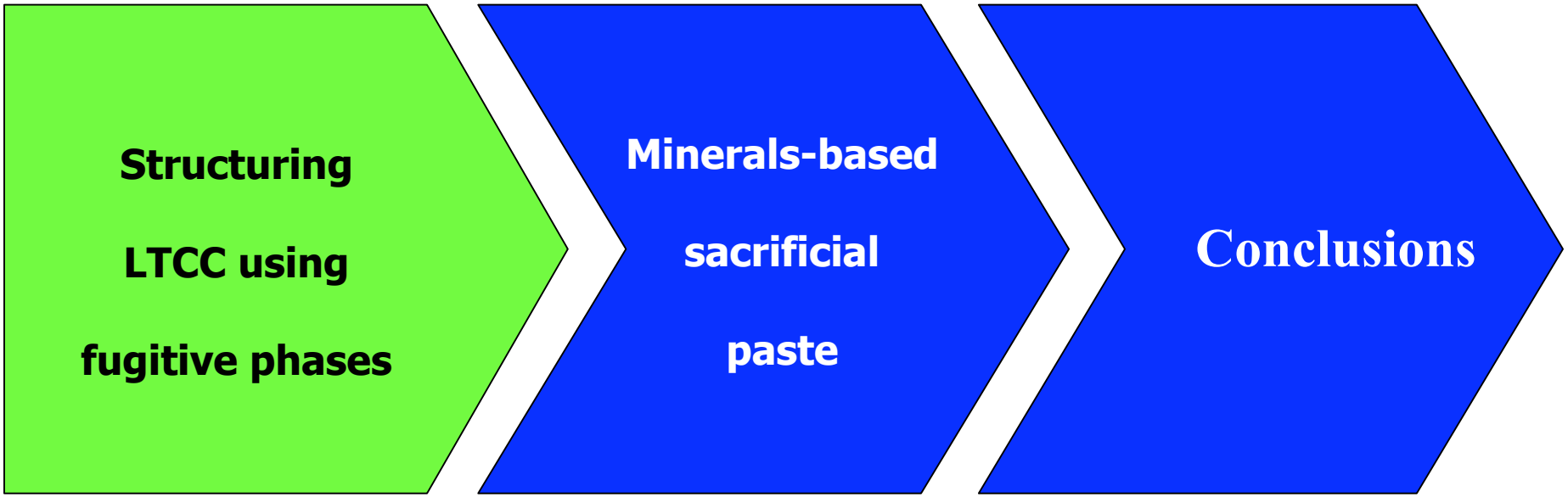

→Comparison of tech-niques

→Why minerals-based sacrificial pastes?

→Preparation of pastes

→Fabricated structures

→Results & discussions

→Next steps

Why fugitive phases for structuring?

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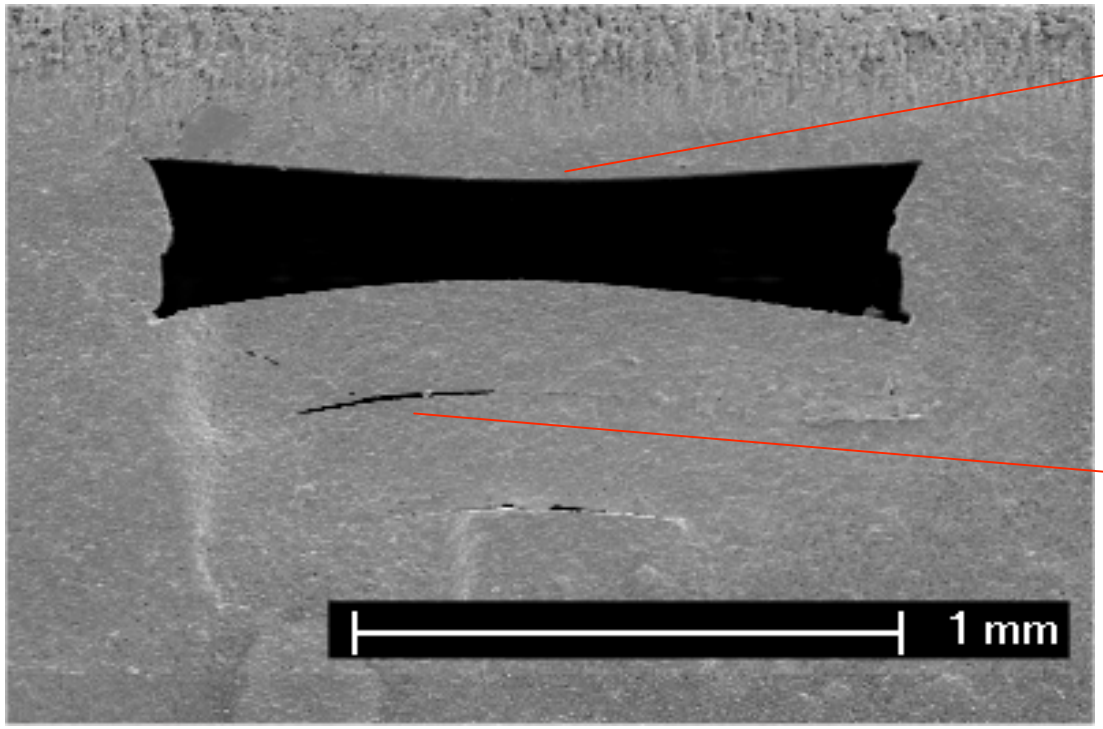
Unsupported cavities during LTCC sintering



Sagging due to lamination or glass softening during LTCC sintering

Delamination due to poorly transmitted lamination pressure

Fugitive phases - overview

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Fugitive phases (below) can be removed during/after LTCC sintering.

1. Precious metals (Au, Ag, …) → expensive
2. Glasses → reactivity with LTCC
3. Graphite tapes → lack of precision, difficulty of processing
4. Graphite sacrificial pastes → narrow processing window
5. Minerals-based sacrificial pastes → ?

Graphite sacrificial pastes (1/2)

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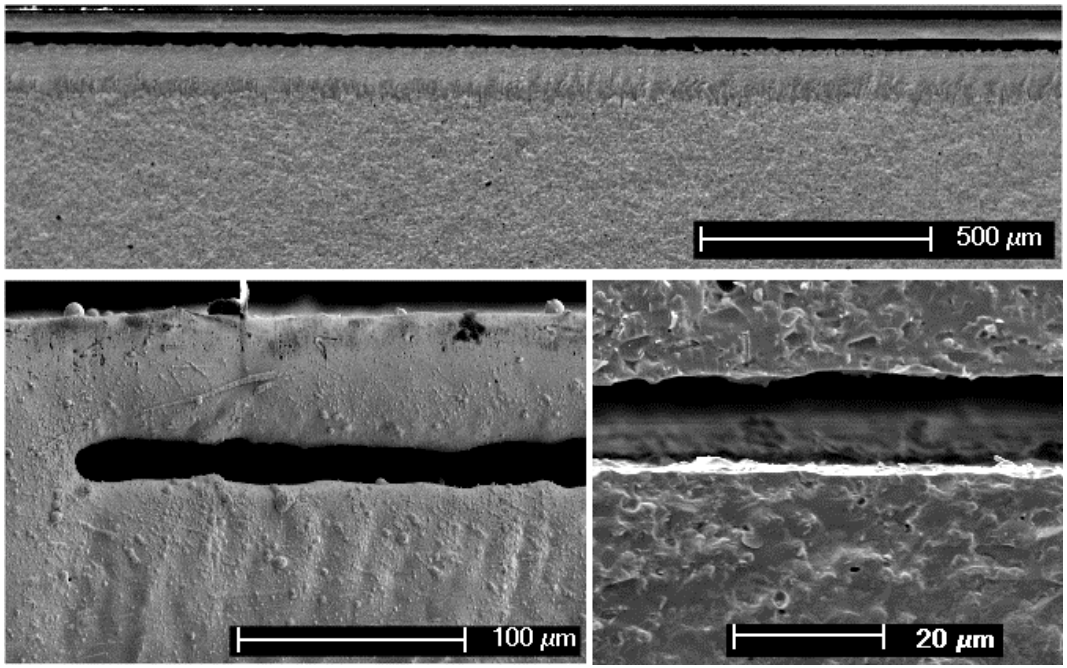
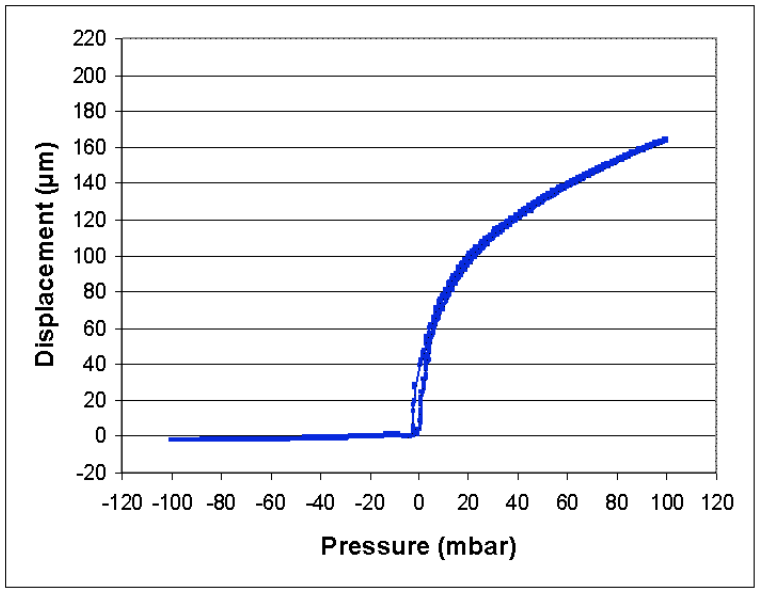
In spite of its demonstrated performance,



-sag-free, well-integrated micro-fluidic channels



-Hystersis-free, pressure sensitive LTCC membranes (displacement up to 200 µm at 100 mbar)

Graphite sacrificial pastes (2/2)

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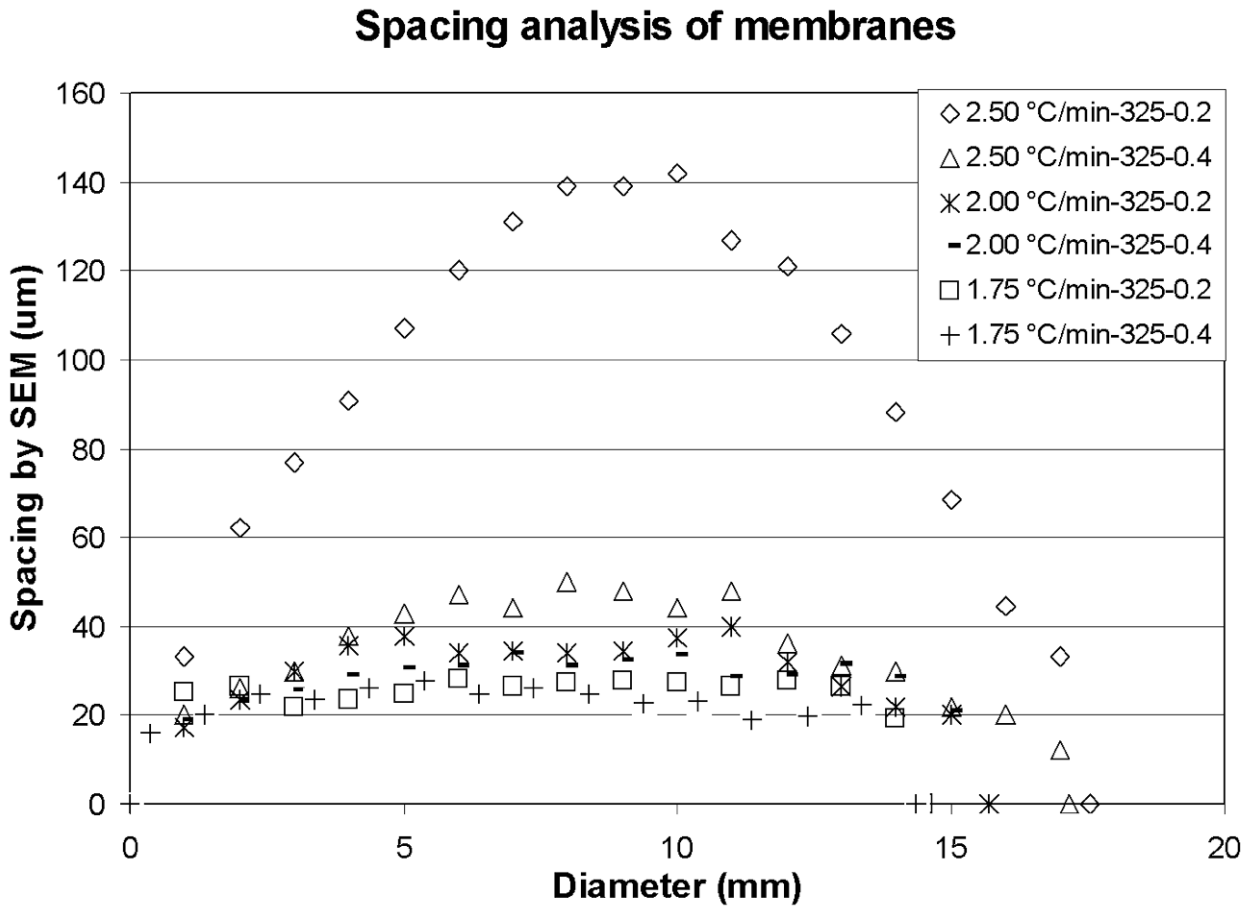
it has a narrow processing window, due to:



Minerals-based fugitive phases (1/2)

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Idea: Preparation of a « permanent » fugitive phase, which
i. can be screen-printed on LTCC,
ii. remains intact until the end of sintering,
iii. can be removed using strong acids after sintering

How: By mixing

Refractory material(s) ($CaCO_3$, $MgCO_3$, etc….)	+	**Low melting glass** (H_3BO_3, etc…)
high T_m (> 2400 °C)		melts at ca. 400 °C

and thus, creating a consolidated state by the porous refractories filled by the glass melt at 900 °C.

Minerals-based fugitive phases (2/2)

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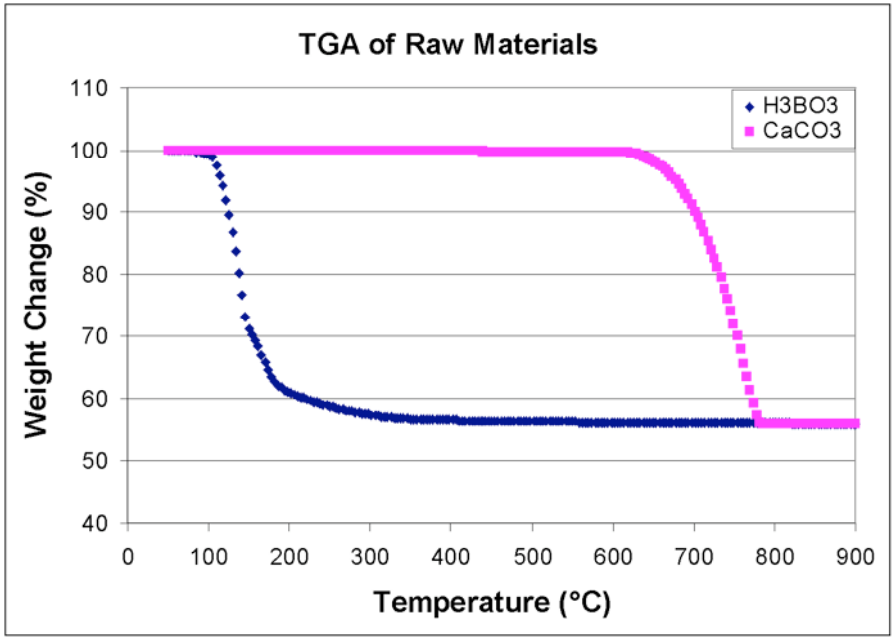

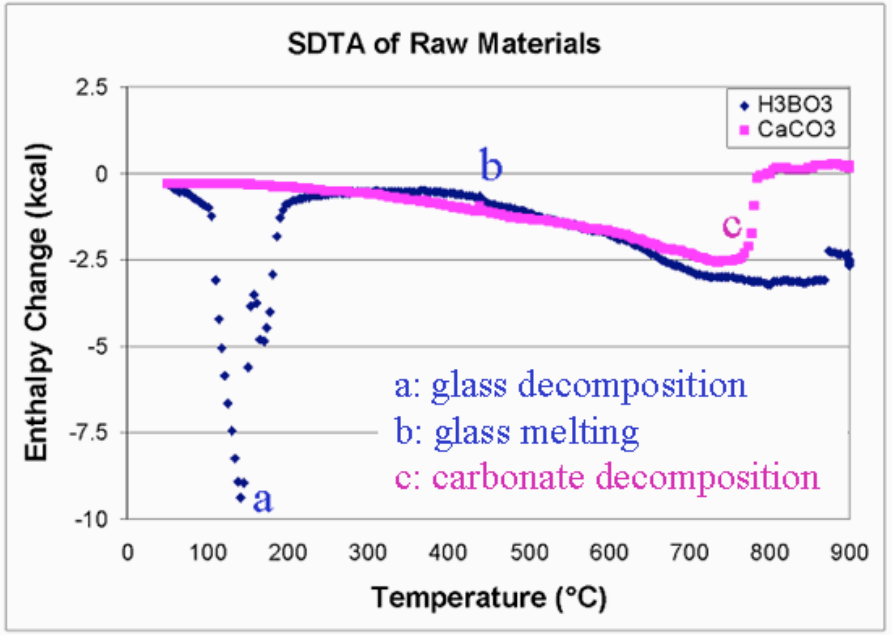

Critical temperatures in light of TGA & SDTA are:

$2H_3BO_3 \rightarrow B_2O_3 + 3H_2O$ (~ 300 °C)

$CaCO_3 \rightarrow CaO + CO_3$ (~ 600 °C)

Outline (2/3)

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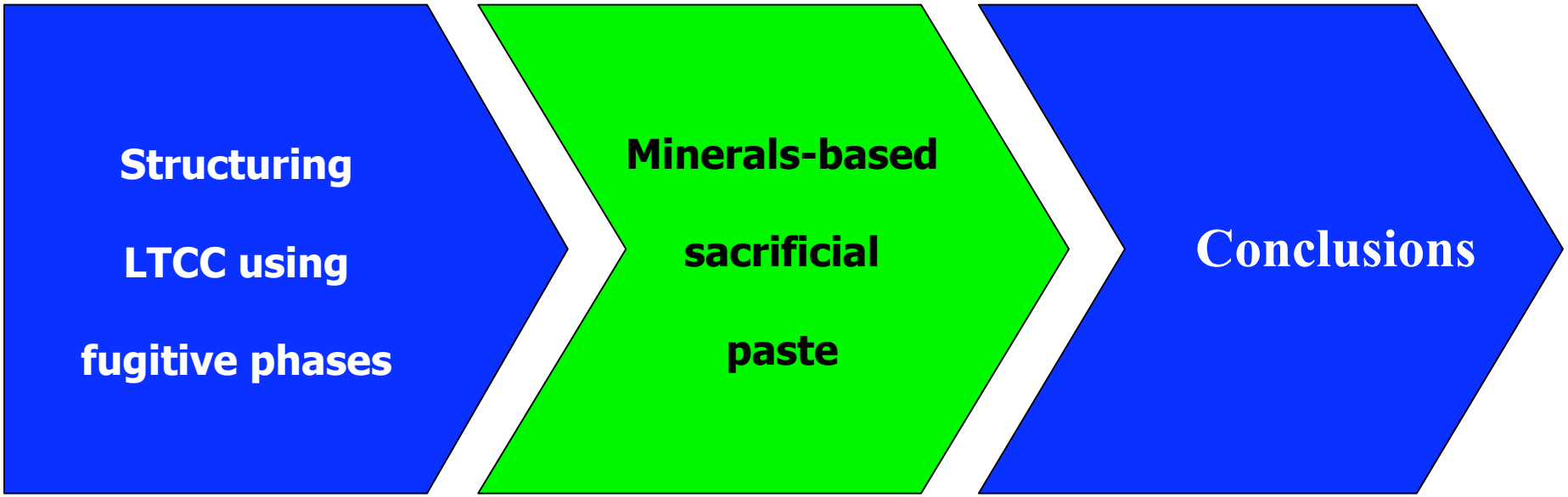

→Comparison of tech-niques

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Preparation of pastes & processing (1/2)

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Pastes are prepared according to the following procedure

1. ($CaCO_3$, and H_3BO_3) powders mixed at stoichiometric ratios and ball-milled in isopropanol for 24h.

10-80 wt. % of B_2O_3

2. Dried and sieved powder mixed with the organic vechicle (below)

42/58 by weight ratio for powder to organics

Product	Function	Supplier	Weight (%)
Ethyl cellulose	Binder	Aldrich, 43,383-7	50.5
Terpineol	Solvent	Fluka, 86480	2.3
Acetyl acetone	Dispersant	Sigma-Aldrich, P775-4	5.3
CB* powder	Sacrificial		41.9

(*): B_2O_3 - CaO powder mixture

Preparation of pastes & processing (2/2)

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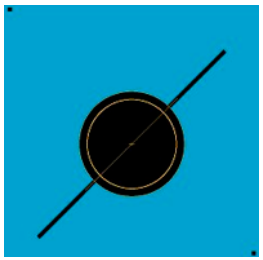
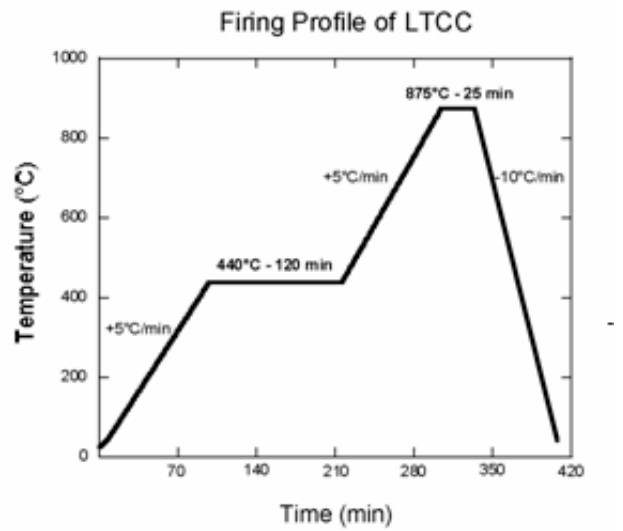
3. Paste screen printed on 254 µm-thick LTCC sheets according to the layout (→)



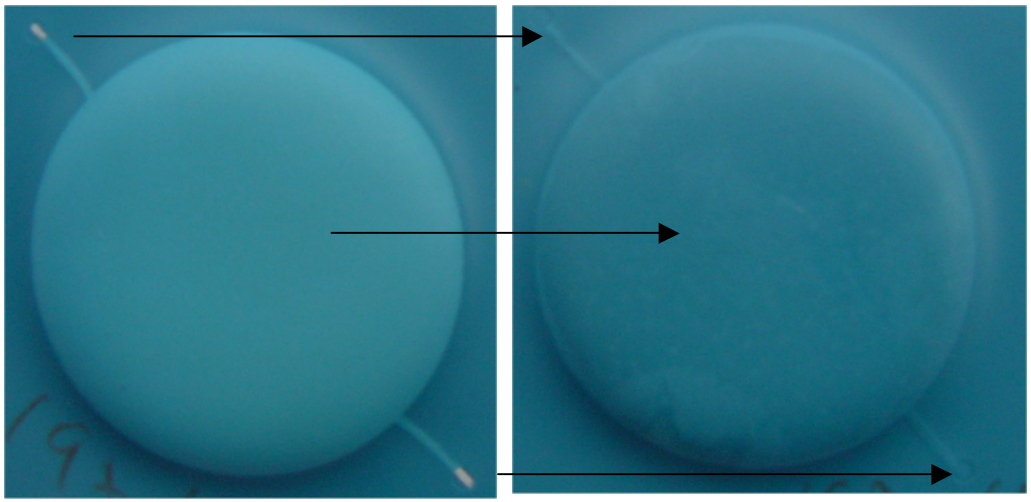
4. Printed layer stacked with a 50 µm-thick LTCC layer and laminated under rubber by 50 MPa of uniaxial pressure (→)

5. Laminated structure fired at 875 °C of peak temperature, following a 2-step firing profile (→)



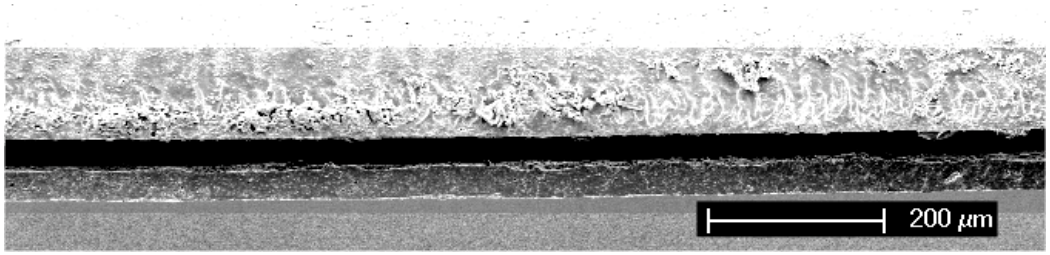
6. The mineral-based layer removed by hydrocloric acid (HCl).

Processing / fabricated structures

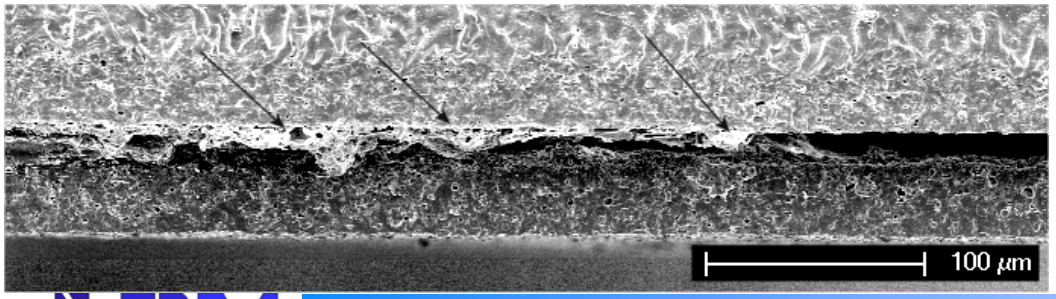


The sacrifical layer dissolved in HCl+US at high rates

HOWEVER, full removal difficult due to capillary forces



Etched and cleaned well



Insufficient removal

Outline (3/3)

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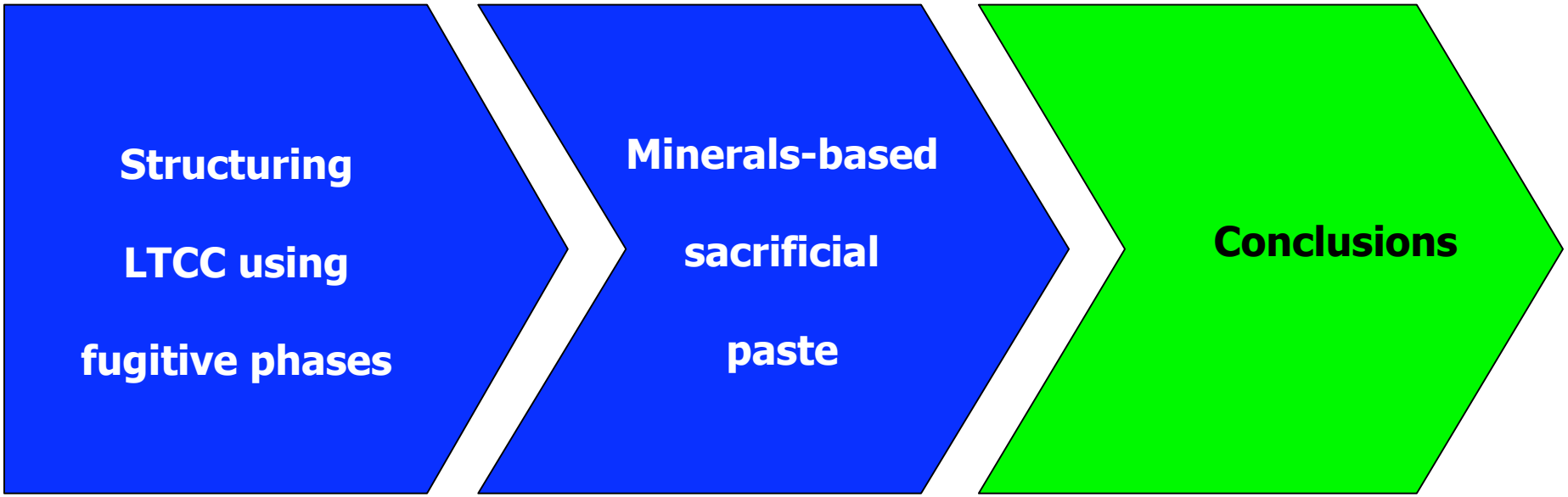

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Discussions & conclusions (1/2)

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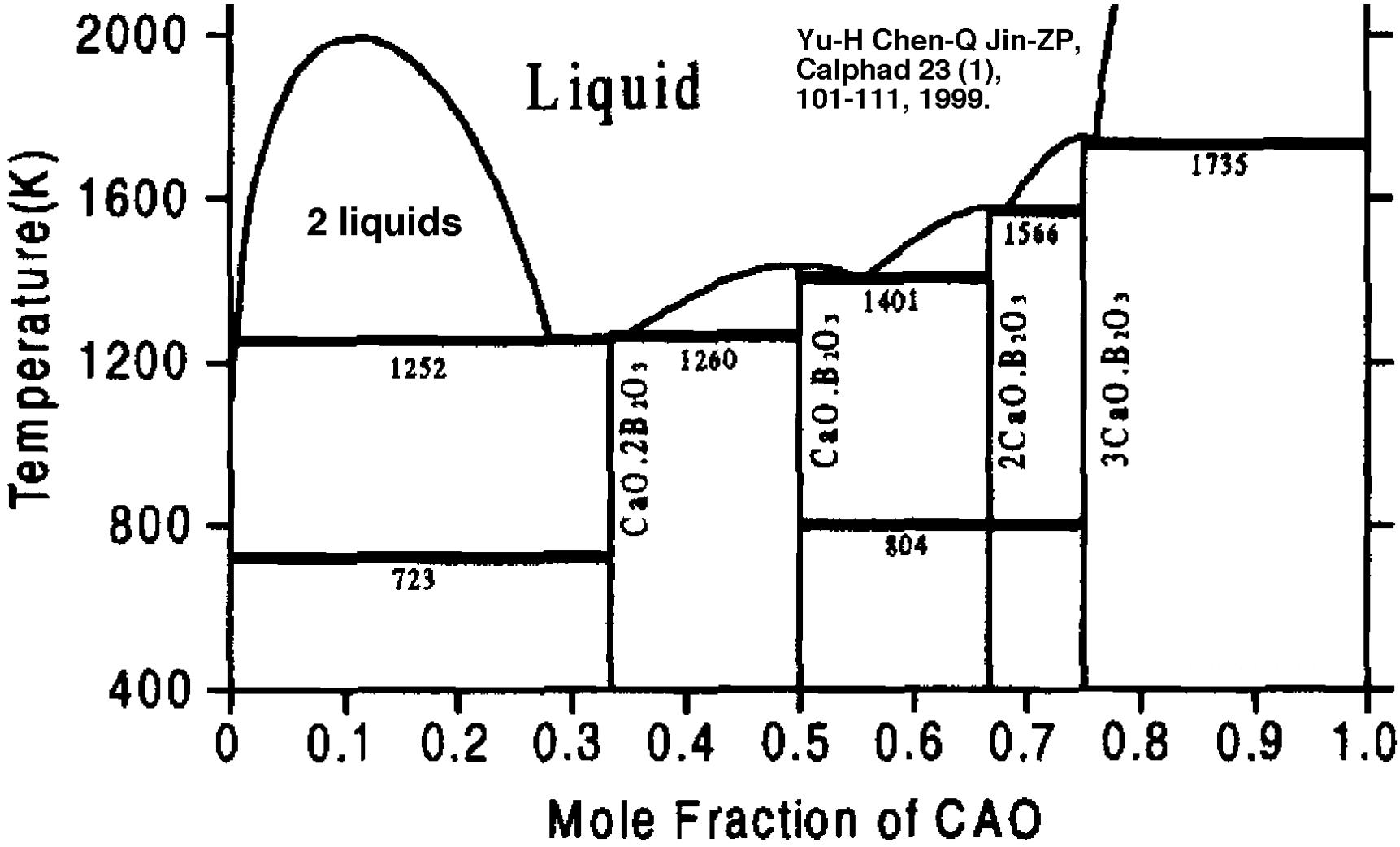
1. The paste remains intact and can be dissolved easily after firing,
2. Sintered paste dissolution by acid is efficient, whereas complete removal of the solution is insufficient,
3. Weight losses of pellets prepared by powders exceeded the losses due to water removal,
4. The major problem is the shrinkage-compatibility of the paste with LTCC,

Discussions & conclusions (2/2)



Next steps

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1. Experiments with H_2O and carbonate-free initial powders,

2. Include other elements such as Na, K in order to match the shrinkage behavior of paste to that of LTCC,

3. Improve the removal of dissolution products

Merci / Danke / Grazie / Hvala

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THANK

YOU !