EMPS 2006 Terme Čatež

Influence of Lamination Parameters on LTCC Shrinkage under Unconstrained Sintering

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Session of Tuesday, May 23, 11:20

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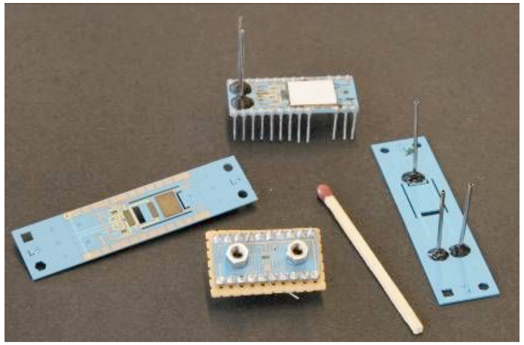
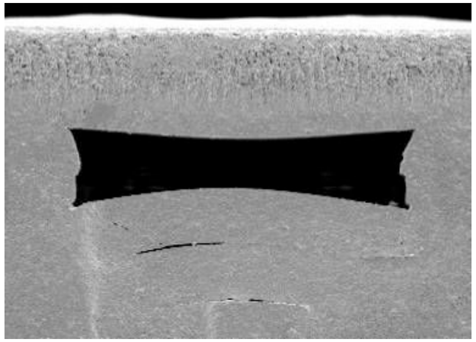
Introduction

For **LTCC microfluidic circuits**,
top manufacturing requirements are:

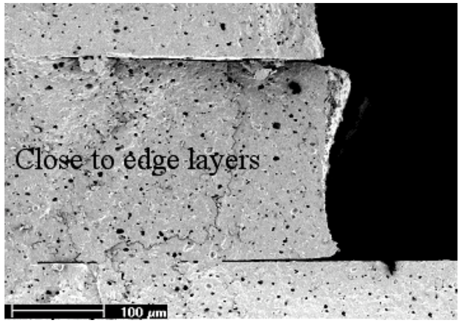


Micro reactors and micro flow sensor

- accurate control of their absolute dimensions

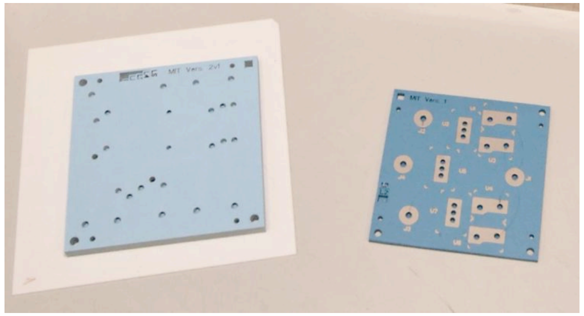


- no crushing of empty cavities



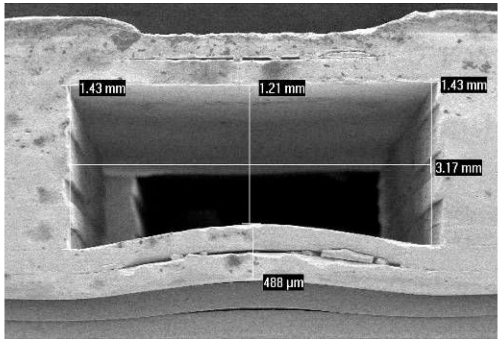
- no delaminations at edges

Our problems

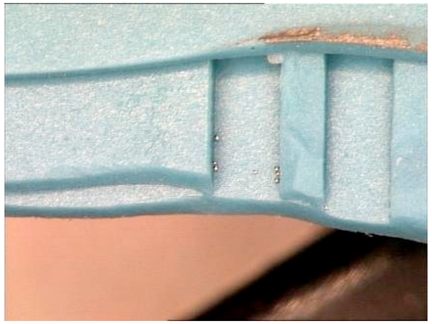


In practice for unconstrained sintering this translates into:

- Variation of final dimensions due to **variability of shrinkage**



- Shrinkage **different (higher) than announced** by manufacturer



- **Crushing of cavities** when following manufacturers' lamination recommendations
- **Layers delaminations at edges** when reducing lamination pressure or temperature

Goals of this study

This study aims to:

- Determine the influence on shrinkage of pre-firing parameters (most obviously lamination)
- Find a model to predict shrinkage
- Find a method to characterise a batch of LTCC
- Shorten the manufacturing process

We will show that *p* and *T* are predominant and obey a linear rule.

The problem of cavity integrity will be the object of future work.

Choice of parameters

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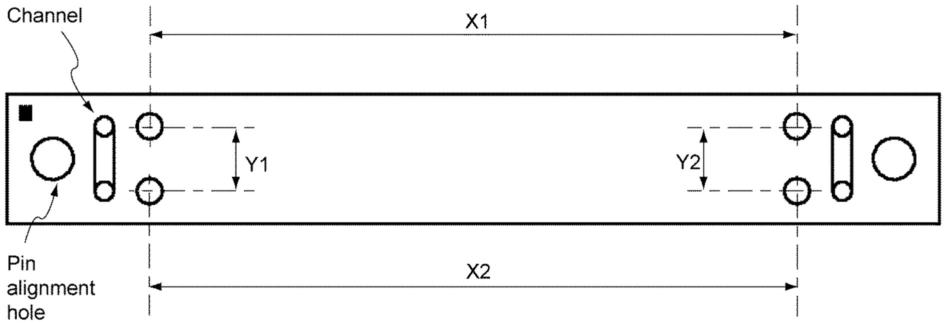
Parameters that can possibly influence LTCC shrinkage:

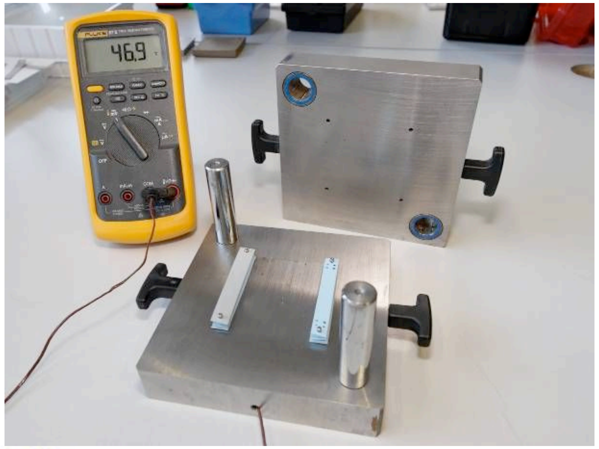
- ageing of LTCC
- method of removing Mylar tape
- pre-conditioning
- blanking method
- type of release tape
- layers stacking method, number of layers *n*
- lamination (type of press, pressure *p*, temperature *T*, duration *t*)
- elapsed times between steps
- firing method and type/flow of firing gas

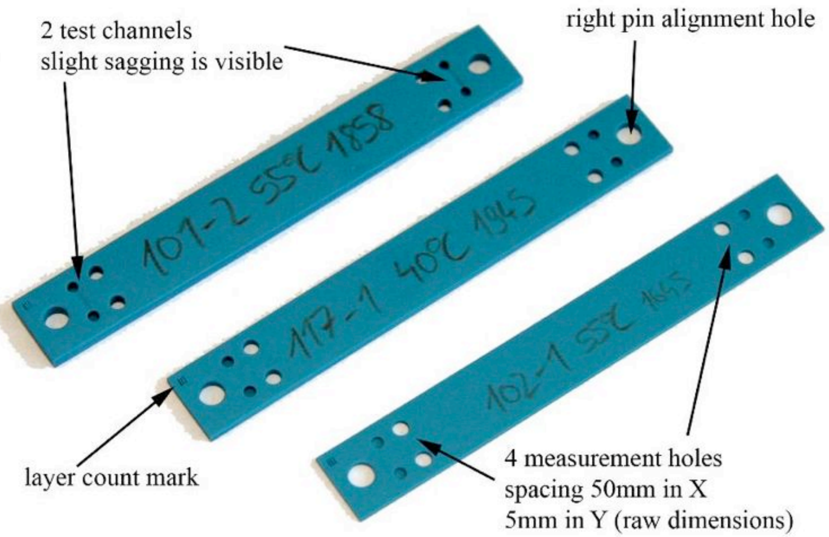
LTCC test samples

Specific 72x10mm test samples have been designed:

- 4 holes for measurements
  - spacing $X_1=X_2=50mm$
  - spacing $Y_1=Y_2=5mm$
- 2 test channels to detect sagging
- 2 holes for pin alignment







Experimental setup – process flow

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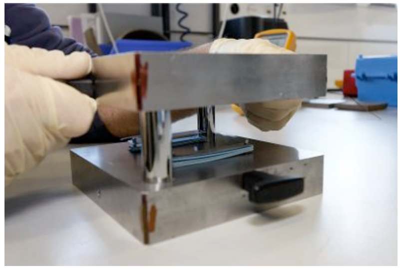
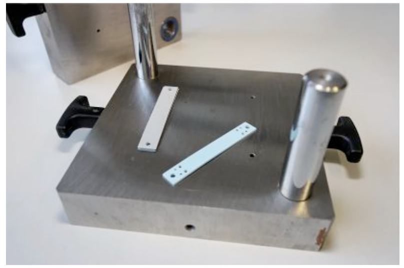
DuPont 951AX 254µm 6”x6” LTCC sheets → Mylar removal + pre-conditioning 30min - 120°C → Laser cutting + air brushing → Optical measurement **State A** →

→ Stacking of *n* layers on pre-heated alignment fixture

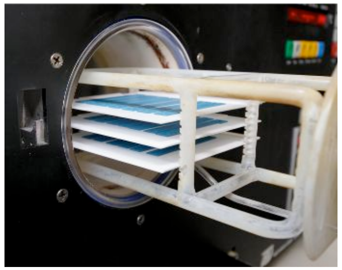


→ 10min rest in pre-heater for temp. equalisation →

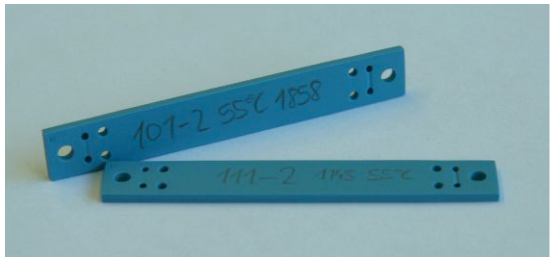
→ Lamination on uniaxial press at *p*, *T* , during *t* minutes → Release tape removal + optical measurement **State B**



→ Firing in box furnace with air 8h - 875°C



→ Final optical measurement **State C**



Experimental parameters – *T*, *t*

Explanations of parameters ranges

- Temperature *T* : 25-55°C
  - Too low (ambient): layers interpenetrate badly
  - Too high (~70°C): LTCC softens too much, cavity crushing
  - *DuPont* parameters cannot be used with our non-standard process, as test samples get too damaged to be measured
- Duration *t* : 5-25 mins
  - In literature we find 5 to 15 mins.
  - Pressure manually hold for first 2 mins, then lever released.

Experimental parameters – *p, n*

Explanations of parameters ranges

	T [°C]	*t* [min]	*p* [bar]
min	25±1	5±0.1	80±7
central	40±1	15±0.1	190±7
max	55±1	25±0.1	300±7
DuPont	70	10	206

- Pressure *p* : 80-300 bars
  - Too low (<80 bars): gets bad laminations
  - Too high (>300 bars): crushes channels
  - Unequal: leads to trapezoidal samples (precise fixture required)
- Number of layers *n* : 3-9 layers
  - must be ≥ 3 to avoid warpage
  - rubbing between LTCC layers could influence shrinkage

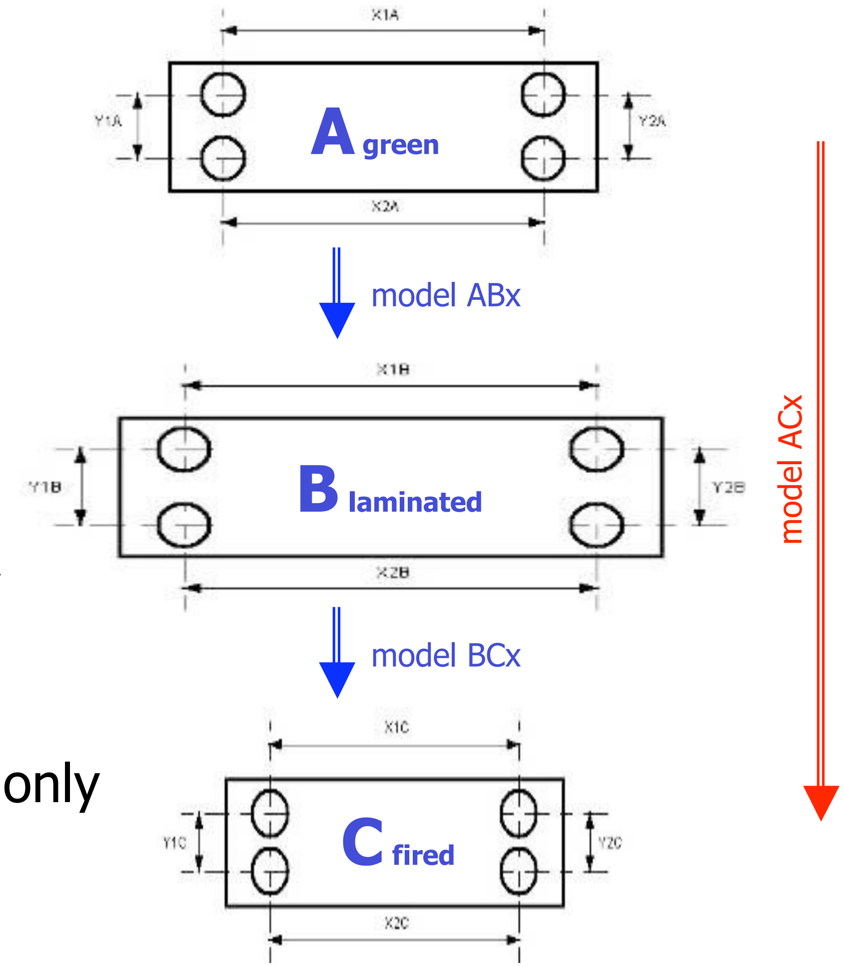
Design of Experiments (DOE)

- Linear Full Factorial Design,
  with central point and interactions

- Focus on variations in X
- Y only for anisotropy estimations

- Initially two sub-models: ABx & BCx,
  but lamination ABx did not output
  relevant information

⇒ We confine the study to ACx model only



Results 1) influence of parameters, ACx

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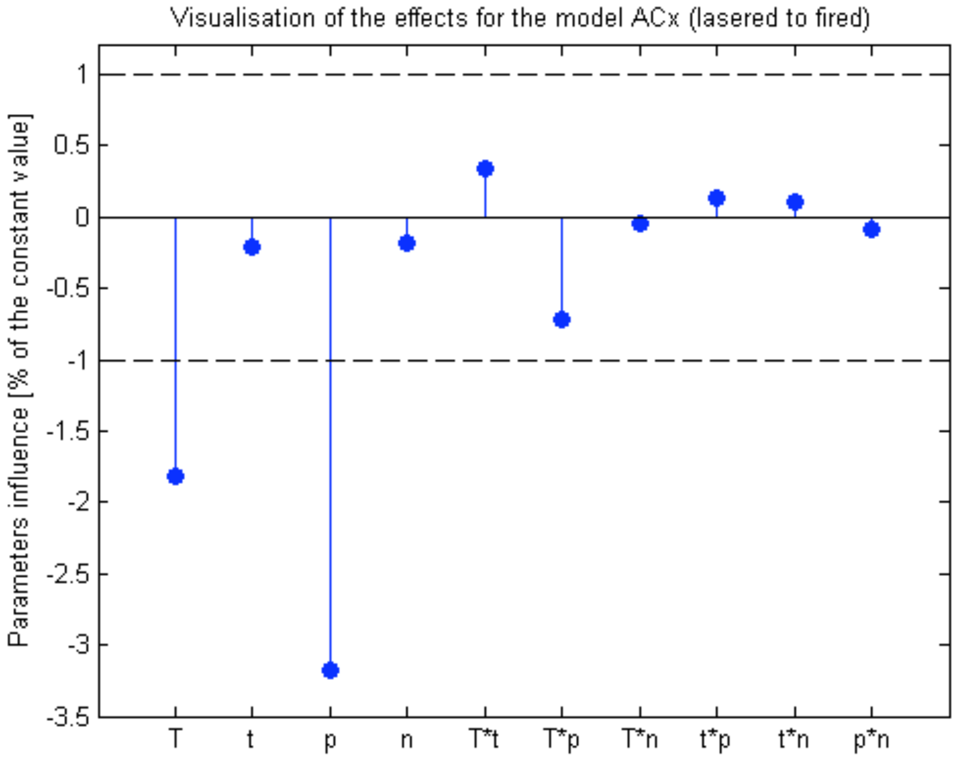
Relative influence of parameters and their interactions in regard to the constant value (13.48% of shrinkage)

- *p* -3.2%
- *T* -1.8%
- *T·p* -0.7%
- *t, n* no big role
- other no big role or negligible



⇒ More pre-densification at lamination lessens the shrinkage (neg. coeffs)

⇒ Simplifications can be made: only *T* and *p* retained (parameters >abs(1%))

Results 2) *T* and *p* only, ACx

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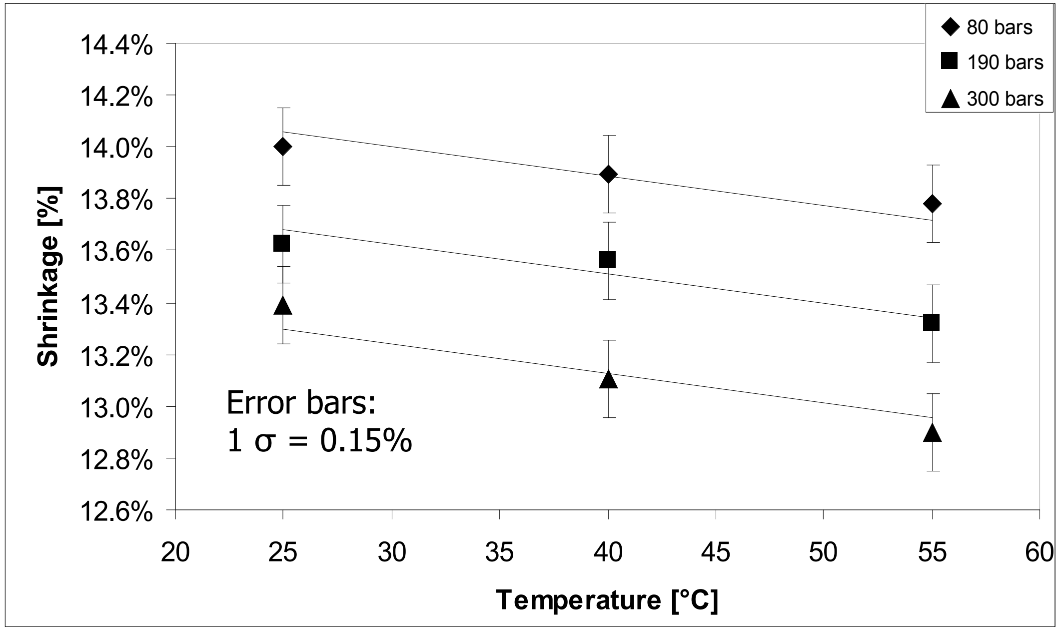
Second run of tests,
t = 5 mins, n = 3 layers.
DOE composite design, N=2



- Clearly a linear relation, but only works in our limited range of parameters
- Relative broad range of shrinkage: from 13% to 14%
- Variability between experiments of same parameters is 2x to 5x bigger than variability between two same samples fired at once
  ⇒ operator variability
  ⇒ LTCC inhomogeneities

Results 3) model of shrinkage

$$f_{ACx}\left[\%\right] = 14.62 - 1.13 \cdot 10^{-2} \cdot T - 3.45 \cdot 10^{-3} \cdot p$$

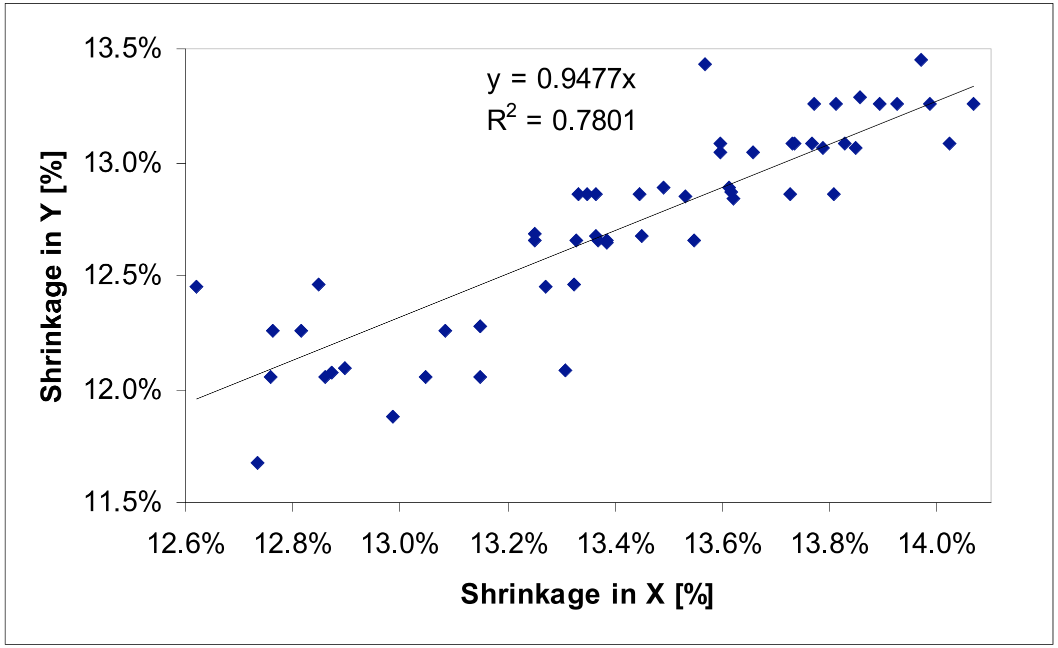
- Formula for our LTCC batch, between 80-300 bars, 25-55°C.
- Good Fischer P-factors for model ($3 \cdot 10^{-12}$) and *T* , *p* (10^{-4} to 10^{-6})
- No *T·p* interaction retained (P-factor 0.63)
- Comparison of model with data from *DuPont* : with *T* = 70°C and *p* = 206 bars, shrinkage = 13.11% instead of 12.5%
- It confirms our expectations, but we must be careful:
  - above 55°C binder properties are expected to become nonlinear
  - *DuPont* recommends 10 mins and we used 5

Results 4) anisotropy

All second runs displayed

Y-shrinkage ≈ 95% X-shrinkage

1 σ = 1.55%



- Linear tendency is observed

- High scatter in the low shrinkage area, especially for 55°C – 300 bars points

- R^2 for this model not satisfactory, experiment with same distances in Y as X should be carried out

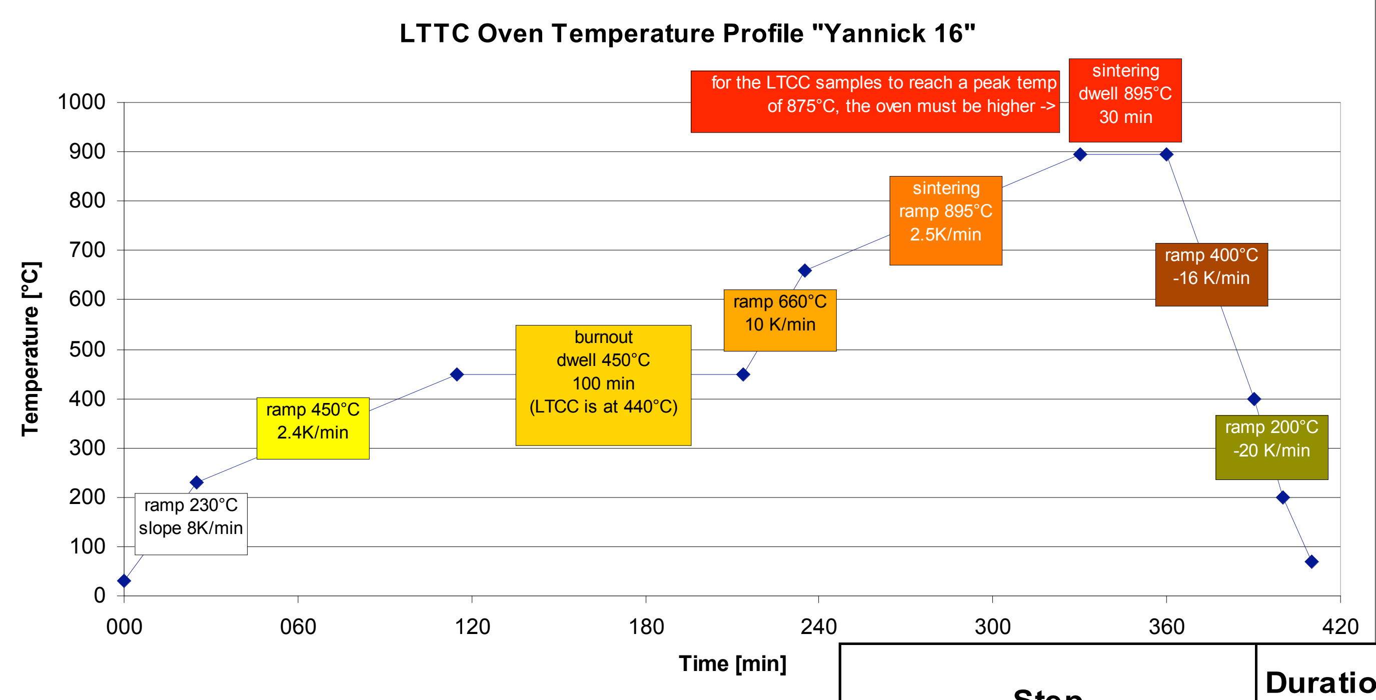
Conclusions

- Non-negligible influence of **lamination** on shrinkage of LTCC unconstrained sintering
- Pressure and temperature are most significant
- Influence of duration and number of layers can be neglected
- Good linear model (P-factor $3 \cdot 10^{-12}$)
- Good process repeatability: 0.15% (usually 0.2-0.3%)
- Results tend to confirm that *DuPont* data is too low: we get 13.11% of shrinkage instead of 12.5%
- Manufacturers could use our method to characterise the shrinkage
- Better understanding of shrinkage, but cavity integrity is still unsatisfying ⇒ new methods of lamination must be sought.

The end

Thank you for your attention!

Annexes – air furnace temperature



	Step	Duration [h:min]	Total time [h:min]	Final temp [°C]	Slope [K/min]
1	Fast ramp	00:25	00:25	230	8
2	Ramp to **440**°C	01:30	01:55	450	2.4
3	Burnout dwell 100 mins	01:39	03:34	450	0
4	Fast ramp	00:21	03:55	660	10
5	Sintering ramp to **875**°C	01:35	05:30	895	2.5
6	Sintering dwell 30 mins	00:30	06:00	895	0
7	Natural furnace cooling	00:30	06:30	400	-16.5
8	Fast cooling	00:10	06:40	200	-20
9	Back to ambiant	00:10	06:50	70	-13