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# Editorial | Uvodnik

Dear Reader,

Year 2012 is running out. Last December days bring festive atmosphere in each home, office or research laboratory. Let last December days bring joy and peacefulness to all. It is the time to look back to the whole 2012, but also to look ahead and make plans for the coming year.

In the last December days Society MIDEM has lost an outstanding member Professor Marija Kosec. After several months of fight against uncurable illness Professor Marija Kosec passed away on 23 Dec 2012. She was a wonderful person, scientist, innovator, leader and mentor. She had been the head of Electronic Ceramics Department of Institute »Jozef Stefan« and led the Centre of Excellence NAMASTE, member of several committees, societies and two academies: Slovenian Academy of Engineering and World Academy of Ceramics, and recipient of several national and international awards. She is past president and past vice-president of the Society MIDEM (Society for Microelectronics, Electronic Devices and Materials) and the Chairperson of the MIDEM 2012 – the 48<sup>th</sup> International Conference on Microelectronics, Devices and Materials with the Workshop on Ceramic Microsystems that was held in September 19-21, 2012 in Otočec, Slovenia.

For more on her work, you can read In memoriam of Prof. Kosec at the end of this issue or browse VideoLectures.net (an EU supported project of the Institute "Jozef Stefan"), where nine of her lectures are posted.

Marija Kosec was named Ambassador of Science of the Republic of Slovenia in 2003, but she had been acting as ambassador of our society since 1989, when she became a vice-president. She was a bright star on the sky of Slovenian Science, a bright star that will keep shining on our society. We will keep a lasting memory of her.

This issue brings seven papers contributed from distinguished invited speakers at the MIDEM 2012 Conference that were selected by co-chairpersons Prof. Marija Kosec and Darko Belavič to present the state-of-the-art achievements from the broad field of Ceramic Microsystems.

Merry Christmas and a Happy and Prosperous New Year!

Marko Topič Editor-in-Chief

P.S.

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Informacije (MIDEM

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# Innovation steps towards a novel and cost efficient LTCC packaging technology for high end applications

Franz Bechtold

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**Abstract:** The emerging of a new MEMS, MOEMS and BioMEMS in the field of Sensors and a new GaN based generation of RF semiconductors in the field of RF Communication create new challenges for the packaging technology. In order to provide adequate packaging solutions for high end applications, manufacturers today are requested for high flexibility, excellent performance and reasonable costs.

Requirements with respect to advanced circuit functionality and enhanced thermal management, low dielectric losses, low parasitic effects and crosstalk, SiP integration, thermal management, miniaturisation and reliability have to be fulfilled. Today LTCC provides a proven packaging solution for Standard MEMS, GaAs devices and MCMs. increasing frequencies; increasing power and increasing functionality of the systems are the driving forces to investigate into novel packaging solutions.

This paper describes the different steps how LTCC technology can cope with these requirements. Based on a comparison of the different available LTCC systems and a benchmark between them, the packaging performance of several LTCC systems is investigated in detail. Technological features, relevant for miniaturisation, System in package integration and RF performance, like cavities, conductor line resolution, via diameter and flatness are considered. Process capability, relevant for thermal management, SiP integration and hermeticity like brazing of heat sinks, integration of heat pipes, integrated capacitors and resistors are analysed.

The impact of novel and low cost materials for metallization, heat sink and brazing will be evaluated with respect to weight and cost. Compatibility with RoHS and REACH regulations will be discussed. The progress beyond the state of the art will be demonstrated by packaging solutions recently developed for industrial applications and in the frame of research programmes.

Keywords: LTCC, sensors, RF packaging, System in Package, reliability

# Inovativni koraki proti novi in ceneni LTCC tehnologiji pakiranja za visokotehnološke aplikacije

**Izvleček:** Združevanje MEMS, MOEMS in BioMEMS na področju senzorjev z RF polprevodniki na osnovi nove GaN tehnologije predstavlja nove izzive za tehnologijo pakiranja. Visoka fleksibilnost, izjemne lastnosti in sprejemljiva cena so zahteve proizvajalcem za izdelovanje ustreznih rešitev pakiranja visokotehnoloških izdelkov.

Današnje LTCC predstavljajo preizkušene rešitve za pakiranje standardnih MEMS, GaAs elementov in MCMjev. Povečanje moči, funkcionalnosti in frekvenc zahtevajo razvoj novih rešitev.

Članek opisuje različne korake rešitev teh zahtev s pomočjo LTCC tehnologije. Različni LTCC sistemi so medsebojno primerjani in njihove lastnosti natančno raziskane. Opisane so tehnološke značilnosti pomembne za proces miniaturizacije, integracije sistemov v ohišje in RF lastnosti. Analizirane so sposobnosti procesa termičnega upravljanja, SiP integracije in hermetičnosti.

Vpliv novih in cenenih materialov za metalizacijo, hladilnike in spajkanja bodo ocenjene glede na težo in ceno. Opisana bo združljivost z RoHS in REACH regulativami. Predstavljene bodo napredne rešitve novega pakiranja za industrijske aplikacije v okviru raziskovalnih programov.

Ključne besede: LTCC, senzorji, nizke izgube, nizka teža, RF ohišja

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## 1. Introduction

New microsystems, microsensors and microelectronic devices like MEMS, MOEMS, BIOMEMS and GaN based high power/high frequency semiconductors are emerging. These devices show an outstanding performance with respect to miniaturisation, functionality and power. Adequate packaging solutions, encapsulating heterogeneously the different components have to take into account that some of these devices are sensitive to environmental conditions like thermal cycling, heat dissipation and humidity. In addition good electrical stability and good EMC performance together with excellent high frequency and high power performances have to be obtained and hermetic or quasi hermetic packaging is requested in different cases. These requirements create new challenges to the packaging technology with respect to electrical performance, SiP integration, thermal management and reliability.

# 2. State of the Art LTCC Packaging solutions

Today LTCC provides an established and proven packaging solution for automotive applications, mobile phone circuits, industrial sensors; GaAs based MMICs and RF MEMS. Passive functional elements like miniaturised antennas, filters, couplers, temperature sensors, heating elements, resistors, capacitors and inductors can be monolithically embedded. Depending on the power dissipation and the reliability requirements those packaging solutions cover the whole range from low power quasi-hermetic packages to high power near hermetic packages and fully hermetic packaged high power MMICs. Glob top and transfer moulding protection, adhesive bonded heat sink and cavity shielded MMICs as well as brazing technology for heat sink and ring frame attachment are used. The type of packaging chosen is related to the lifetime, the reliability and the volumes of the different application.

# 3. Packaging solutions in the low volume industrial market

The low and medium volume market for LTCC is mainly represented by customized packages in the field of industrial sensors, medical devices and high end rf modules for telecommunication and satellite communication. Driving forces for all of those applications are the technical performance at acceptable cost for the individual customer solution. In the following example (Fig.1) the complete package of an x-ray detector is shown.



Figure 1: KETEK: x-ray detector

The key benefits with LTCC is the high miniaturisation, the capability of thermal management which includes a channel for heat transfer and thermal vias for power dissipation, the sspectroscopic neutrality and the full Hermeticity with 0 outgassing.

In Fig. 2 different customized packages for X-Ray detectors and pressure sensors demonstrate the benefits of specific shapes; several wire bond levels, integrated fluidic channels and the capability of dedicated material systems.



Figure 2: VIA: Customized sensor packages

Key benefits shown are specific shapes, several wire bond levels and Integrated fluidic channels Dedicated material systems

# 4. Packaging solutions in the high volume automotive market

Cost together with reliability and Miniaturisation are the driving forces in the automotive business. LTCC in Germany's automotive market today is a business of about 250 Mio €. Typical under the hood applications are Antiblocking, Gear control or Steer by wire systems.



**Dickschicht Substrate** 

**DBC Substrate** (Direct Bonded (opper)

Figure 3: TEMIC: Technology platform

All of them request heterogeneous integration of highly miniaturised signal electronics together with thickfilm and DCB high power substrates into the same package.

(Low Temperature Cofired Ceramic)



Figure 4: TEMIC: Gear control unit

# 5. Packaging solutions in the high volume mobile phone market

In the high volume market of mobile phones a fast evolution of miniaturisation using LTCC packaging technology can be observed.

Front end (Fig. 5) is realized today in LTCC. The key benefit of LTCC is the possibility to integrate passive RF functions at substrate level. Using a material system which consists of two different dielectric materials, one for signals with K= 8 and the other with K= 20 (the so called electronic ceramic) enables to integrate a number of such functions like filters, baluns, matching networks and others.



Figure 5: Epcos: Schematic of a LTCC frontend,

Fig. 6 depicts the top view of a dual-band WiMAX LTCC module. Active components like PA, switch and even some power management (PM), functionality are soldered on the top of the module while the RFIC is connected by means of wire bonds.



Figure 6: TDK-EPC: WiMAX SiP

The worldwide smallest All-in-one Frontend-Modul for Bluetooth- and WLAN-application has been published by Epcos 2008. With a height of 1,4mm it requests only a small area of 4,5 x 3,2 mm<sup>2</sup> on the PCB. This module integrates the power amplifier, the switch, the receivebalun and the bias network with EMW protection. Furthermore, the integrated coexistence filter allows simultaneous operation of WLAN and Bluetooth having all standards of mobile communication and realises all requested functions between WLAN and Bluetooth.



Figure 7: Epcos D 601: The world smallest All in One Module

The most actual development is an advanced frontend, based on the co-design of Frontend and Antenna by combining flex foil and LTCC. This module combines the advantages of LTCC and Flexible board technology and results in a game-changing 16-band antenna, including Japanese frequency bands in the 1.5 GHz range. It is a single hardware-serving operator worldwide.



Figure 8: TDK-EPC: Advanced multi-feed RF front-end

# 6. RF Packaging solutions in other applications

The telecommunication market requests also for high end solutions at medium volumes. High power T/R modules, antenna- and phase shifting modules, RF-MEMs Switches are widely applied in active antennas for satellite communication and radio links. Radar detection systems are used in defence applications. Radar sensors have been successfully introduced in the automotive business and industrial applications are following. The following pictures show a 60GHz-band 500-Mpbs transmitter and receiver multi-chip modules (MCMs), in which MMICs and filters are mounted into a LTCC package using flip-chip technique. A Lid protects the IC's and the MCMs are directly bonded with printed wiring boards using ball grid array technique, achieving connections for signals and biasing.



Solder Ball(BGA)

Figure 9: NEC: Schematic of the NEC 60 TR module

The transceiver is applied to the IEEE1394 wireless adapter, which demonstrates 17-mcommunication distance in line of sight.



Figure 10: NEC 60 TX and RX Module

LTCC is offering again numerous advantages like the rf performance and the capability of passive integration. Another one is the design flexibility combined with the approach of modularity. Ericsson has demonstrated how to develop a number of T/R modules working at different frequencies from 15 to 39, using the same or similar RF building blocks for the realisation of the circuit. Filters, waveguide transition and other RF functions were successfully integrated at substrate level together with buried resistors. The other advantage is the high reliability performance.



High integrated Microwave Transceiver Modules RF module in LTCC

**Figure 11:** Ericsson: Modular kit of LTCC T/R modules, 15 to 39 GHz

LTCC circuits perform a lifetime of more the 20 years, they are stable under harsh environment and they are capable for full hermeticity according to Mil Std. Those substrates can be sourced from foundries like VIA on the free market in Europe.



Figure 12: Thales: 3-bit phase shifters for phased array antenna

For space and military applications hermeticity is a must. Weight plays an important role and also cost is a key issue for success. Efficiency in packaging and miniaturisation in order to reduce cost, weight and size are key benefits of the LTCC technology.

According to the high energy requested for transmission, power dissipation and thermal management are integral part of the packaging requirements and components like heat sinks and ring frames as well as the attachment of those elements to LTCC are in the focus of this paper.

The following picture shows the TNO design of a state of the art packaging solution for a TR module working at K- band for nautical application.



Figure 13: TNO: Design of a LTCC Transmit Receive Module

The package contains a rather complex stepped cavity structure for MMIC, Circulator and IC integration. The LTCC is adhesive bonded to the Molybdenum heat sink and then populated with SMT and chip and wire technology.

A further step towards reliability has to be made if full hermeticity of the packaged dies is required. This is the case in space and defence radar applications. An example is given in the following package of a TR module for phased radar antennas. Molybdenum heat sink and Kovar ring frame are AuSn brazed to the LTCC substrate. This package type shows extremely good thermal and reliability behaviour and it fulfils the MIL specification for hermeticity (>5x10<sup>-8</sup>atmcm3/s)



Figure 14: EADS: LTCC T/R Package

Another approach to achieve full hermeticity and good heat dissipation is shown in the following example, in which a Kovar frame is AuSn brazed to achieve hermeticity and a BGA is used on the backside for PCB assembly.



Figure 15: SELEX: RF package Kovar ring and BGA

## 7. Future system requirements

Increasing frequencies, power and functionality of systems are the driving forces to investigate novel packaging solutions. In the following pictures typical T/R modules for x-band and wide band are shown.



Figure 16: Thales: Block Diagram Wide Band T/R module

Typical functions and sizes to be integrated are:

- HPA: 20 mm<sup>2</sup>
- Driver: 5 to 10 mm<sup>2</sup>
- LNA: 5 mm<sup>2</sup>
- MEMS: 2 mm<sup>2</sup>
- Decoupling capacitors and others about 30 mm<sup>2</sup> in total
- Temperature sensor

In order to have a smart package, the internal room for components is thus typically estimated from 100 to 250 mm<sup>2</sup> for wideband and 150 to 400mm<sup>2</sup> for x-band. The developed package must be compatible with a cold plate cooled by fluid.

Power to be dissipated is up to 80 watts peak dissipated power, pulsed with a pulse time from 1 to  $20\mu s$  associated to a duty cycle of up to 20%.

Materials with excellent thermal conductivity are required. These materials must be compatible with high temperature gold tin brazing. Return loss of one microwave transition must be better than – 25 dB and the isolation between the antenna access and the LNA access must be better than -80 dB. The isolation between biasing and /or control ports must be typically 50 dB, which is typically realised by a metallic wall.

As sensitive devices are encapsulated a good hermeticity must be by enclosure obtained. Typically a 5x10-8 Atmcm3/s (air) is required. The package must withstand  $-55 + 125^{\circ}$ C 500 cycles without lack of hermeticity or electrical performances degradations.

### 8. Steps to cope with these requirements

The expertise in the technology together with the relevant application know how of the customer is the backbone of the small and medium sized European LTCC Foundries like VIA in order to service the free mar-

# 9. Understand materials and their

ket with high flexibility and medium to low volumes.

properties



**Figure 17:** VIA: LTCC RF packages transition from Du-Pont 951 towards DuPont 9K7.

# 10. Overview

One of the most mature systems used is DuPont 951. The RF capability of this system is limited to about 10 GHz, dependent from design and application. For RF applications above 10 GHz specific low loss materials are available from different suppliers. RF Properties and characteristics are listed in table 1, in comparison with DuPont 951:

#### Table 1:

	Comme	Commercial LTCC materials for High Frequency			
	951 DuPont	943 DuPont	9K7 DuPont	9K5 DuPont*	A6M-E Ferro
Permittivity	3				
≤ 1 GHz	7,8	7,5	7,1	5,8	5.9+/-0.20
1-20 GHz	7,4	7,4	7,1	5,8	5.9+/-0.20
20-40 GHz	-	7,4			5.9+/-0.20
Dielectric lo	sses tan $\delta$	[10-3]			
≤ 1 GHz	< 2	< 1	< 1	≤1	≤1
1-20 GHz	5	1	<1	≤1	≤2
20-40 GHz	<15	< 2			≤2
Insertion loss [dB inch-1]					
Conductor	Ag	Ag	Ag		
1- 20 GHz	≤ 1,4	≤0,3	≤0,3		

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And in table 2 physical properties and available tape thicknesses, relevant for RF designs, are as given and compared to DuPont 951

#### Table 2:

Physical Property	DuPont 951	DuPont 943	DuPont 9k7	DuPont 9K5	Ferro A6ME
	Value	Value	Value	Value	Value
Unfired thickness [µm]	50 <u>+</u> 3 114 <u>+</u> 8 165 <u>+</u> 11 254 <u>+</u> 13	51 <u>+</u> 4 127 <u>+</u> 9 254 <u>+</u> 13	127 <u>+</u> 9 254 <u>+</u> 14	127* 254*	127 254
X,Y, shrink- age, [%]	12.7 <u>+</u> 0.3	9.5 <u>+</u> 0.3	9.1 <u>+</u> 0.3	9.1*	15.4 <u>+</u> 0.3
Z shrinkage, [%]	15.0 <u>+</u> 0.5	10.3 <u>+</u> 0.5	11.8 <u>+</u> 0.5	11,8*	24.0 <u>+</u> 0.3
Dielectric constant, (40GHz)	7.4	7.4	7.1 <u>+</u> 0.2		5.9 <u>+</u> 0.2
TCE α [ppm K-1] (25-300°C)	5,8	4,5	4,4	4,4*	5,6
Environ- mental		Lead- free	Lead- free	Lead- free	

Large entities like the big Japanese and Bosch are dealing only with high volume markets. Bosch is focussing on automotive and Epcos on the telecom business. They all have their proprietary material systems either provided by external partners or made by themselves. Constrained sintering and plating are key process for cost and performance reasons. All these processes are dedicated to automate high volume production. A summary is given in table 3:

#### Table 3:

Murata	TDK-EPC	Kyocera
K8.8/K15.1	K8/K20	K9,4/K18,7
for telecom	for telecom	for telecom
K7.7 for automotive	Plating	K7,7 Cu
Plating	Pressure assisted	K5,7 Cu
Pressure assisted	sintering	K5,7 Cu
sintering	(0-Shrinkage)	K5,2 Cu
(0-Shrinkage	DuPont 943 for	Plating
	automotive	

It is obvious, that a deep understanding of materials and processes as well as a good knowledge about the application requirements have to meet together in order to provide proper cost efficient LTCC packaging solutions.

# 11. Benchmark DP 943/9K7

In order to understand deeply the different materials, VIA made a detailed manufacturability benchmark between DP 943 and DP 9K7. The impact of thermal coand post processes parameters on RF characteristics and geometrical properties of LTCC was investigated. Radar front ends also including calibration structures and test vehicles were fabricated using Du Pont Green

Tape<sup>®</sup> 943 and 9k7 material systems. The following parameters have been investigated:

- The x- and y- dimensions both after co-firing and re-firing
- Warping of the whole substrate
- Warping of cavities
- Characterization of electrical properties up to 110 GHz
- Influence of re-firing on the dielectric constant.

The LTCC wafer has been a multi-project wafer, having a particularly complicated package combining several features with unfavourable impact on even and homogeneous shrinkage. Uniformity of the shrinkage is constricted by perforations, clusters with more metal content, cavities or cut-outs. The following Figure shows the test vehicle MPW1 to visualize the local differences of metallization.

The left part of the substrate contains 80% of the vias and 70% of the co-fired conductors, while the right side has larger metallization of inner layers and cavities as chip pockets or decoupling features. Both parts of the substrates were measured separately in x- and y-axis in order to quantify possible differences in the local and overall shrinkage. The results are shown in table 4. Shrinkage values are given in % deviation from the mean value. Warping is given in µm of maximum to



Figure 18: VIA: Multiwafer design

minimum value using a 40mm long scanning line. Refiring stability in % expansion after re-firing.

#### Table 4:

Investigation	measurement	DuPont 943	DuPont 9K7
Shrinkage sensitivity	Shrinkage around cavity areas	-0,175%	-0,19%
	Shrinkage in dense metal- lisation area	-0,02 to -0,040%	-0,02 to -0,03%
	Shrinkage in low metallisa- tion area	0,02 to 0,035	0,02 to 0,035%
Shrinkage accuracy	Overall sub- strate area	+/- 0,16%	+/- 0,13%
Planarity sensitivity	Warping around cavity	-3 to 17µm	-3 to 17µm
	Warping in dense metal- lisation area	9-15µm	10 – 14µm
	Warping in low metallisation area	15-22µm	16 – 17μm
Dimensional Stability vs. refiring	Expansion after 3 refirings	0,07-0,11%	0,07-0,11%
	Expansion after 5 refirings	0,14-0,17%	0,13-0,15%
	Expansion after 8 refirings	0,17-0,21%	0,16-0,18%
Dielectric Constant vs. refiring (20-60GHz)	Deviation of the Dielectric constant after 5 refirings	-0,10 to -0,12	-0,02 – 0,02

The 9K7 LTCC material shows excellent RF performance up to 100 GHz and above. Comparing to the previ-

ous generation 943, process stability, higher accuracy in its shrinkage behaviour and RF stability have been improved. Due to these benefits it allows SiP solutions with complex structures and a high accuracy of cofired features.

# 12. Understand the requirements

Table 5 gives an overview of the different systems available concerning the aspects of miniaturisation and process capability, which impact cost and performance. Datas are based on the experience of VIA..

#### Table 5:

Investigation	DuPont 951	DuPont 9K7	Ceram tape	Heraeus CT 700
Available sheet thick-	50			
ness µm	114	127		100
	165			
	254	254	320	320
Shrinkage tolerance %				
Standard	0,6	0,6	0,6	0,8
Minimum	0.3	0.3	0.3	0.5
Cavities	1,0	1,0	1,0	1,2
0-Shrinkage Capability	++	+	++	++
Line width µm	100	100	100	100
Line spaces µm	100	100	100	100
Dimension Stability				
Green, µm	30-70	30-50	<20	<30
Refiring, in %	0,1-0,15	0,1-	<0,1	0,1
		0,15		
Embedding capability				
lines >30µm	+	-	+	+++
resistors	++	-		
high k capacitors	+-	-		

Process sensitivity				
Warpage	++	+	++	-
Waviness	+	+	++	+-
Metallisation impact	+-	-	+-	+-
Maximum density of				
metallisation %	75	75	75	75
Line density				
Width	100	100	100	100
Spaces	100	100	100	100
VIA density				
Diameter µm	100	100	150	150
Distance µm	250	250	250	250
Postfired resistors	+++	+	-	-
Embedded Fluidic				
Channels	+++	++	++	++
Chambers	+++	++	++	++
Bonding				
AI	++	++	++	+
Au	++	++	+-	++
Al heavy wire	++	-	-	++
Cavity bonding				
Al	++			
Au	++	++	+	++
Soldering				
PbSn 95-5	++	++		++
SnAg	++	++		++
Brazing				
AuSn	++	+		
GeSn	++			
Laser processing				
Green	+++	+++	+++	+++
Fired	+-	+	++	+++
RoHS/REACH compat-	In pro-	Yes	Yes	In pro-
ibility	cess			cess
Lead-free	no	yes	yes	yes

In order to provide adequate packaging solutions for high end applications, manufacturers today are requested for high flexibility and excellent performance at acceptable costs. Also RoHS and REACH compatibility have to be considered.

# 13. Introduction and impact of novel and low cost materials

To improve weight and cost, VIA did investigations in alternative materials for heat sinks, brazing and mixed metal wiring. This work is initially focused on the mature DuPont 951 material system and then expanded to the new 9K7 system. The criterions for materials investigated were availability, thermo mechanical properties, weight and cost. All materials and components investigated were introduced on an existing functional RF design. Hermeticity was tested and compared with standard package performance.

# 14. Carbon-fibre Aluminum Composite (CarfAl)

This material is very promising concerning weight, machinability and costs. The following pictures are demonstrating that customer designed heat sink and ring frame elements can be easily realized. Solder experiments have been carried out using the same AuSn performs as they are used with conventional Kovar and Molybdenum Components. The solderability was excellent and no difference to conventional components could be observed. Hermeticity tested was according to MIL Standard at 5x10<sup>-9</sup>, comparable to conventional components.

After Hermeticity tests some defects were identified: small blisters occurred on the plated surface, which could be eliminated at the supplier side by improvements in the plating process.

Figure 19: CarfAl heat sink and ring frame after hermeticity tests



# 15. Solder performs

AuSn brazing using solder performs is well established for high hermetic packages required in space and defence industry. It is obvious that Au is a cost-driving element in these packages. Promising alternatives are high lead containing solder materials, which are widely used for soldering of power devices or sealing of ceramic sensor packages. Additionally SnAg alloyed performs have been investigated..



**Figure 20:** Preform Pb92.5Sn5Ag2.5 on AgPt, CarfAl ring frame

Pre form	Liquid. temp. °C	Pad	Ring	Herm. atm	Result
	AgPt	Kovar	3x10-9	Excellent	
ובר		AgPd	Kovar	3x10-9	Good
BOSr	278	AgPd	Kovar	3x10-9	Good
Auß		Au/AuPd	Kovar	3x10-9	Good
		AgPt	Kovar	3x10-9	Good
2.5	5	AgPt	CarFAL	6x10-9	Bad
287 287 287	AgPd	Kovar	leaky	bad	
	287	Au/AuPd	Kovar	5x10-9	acceptable
		AgPd	Kovar	2x10-9	acceptable
	AgPd	Kovar	leaky	bad	
3.5	5	AgPt	CarFAL	leaky	bad
Ag	221	AgPd	CarFAL	leaky	bad
96.5	221	Au/AuPd	CarFAL	leaky	bad
Sn		AgPt	Kovar	leaky	bad
0.5		AgPt	CarFAL	6x10-9	acceptable
4Cu		AgPd	CarFAL	leaky	bad
Ag0.	228	AgPd	Kovar	5x10-9	good
9.1		Au/AuPd	CarFAL	5x10-9	acceptable
Sn9		AgPt	CarFAL	4x10-9	acceptable

Table 6 shows some promising results:

# 16. Mixed metal LTCC

With respect to LTCC, changing from AuSn to high lead and tin/silver alloys require a change in the thickfilm metallisation beneath. Expensive Au/AuPd metallisation systems shall be replaced by more economic Ag/ AgPd systems or even simple AgPt solder pads. Consequently, Ag shall replace external and internal Au wiring, and AgPd and only pads for wire bonding have to be fabricated with gold. Unfortunately Ag has very low activation energy and tends to migration. Therefore a proper diffusion barrier system has to be introduced in order to avoid silver migration problems.

In the past, diffusion barriers made of AgPd inks were used between Au and Ag conductors. As far as vias are concerned connecting a silver conductor with an Au pad, AgPd transition vias are used. These transition vias show the disadvantage to expand during firing and to create a non-negligible posting of via, which arrives up to 30µm. Those postings are a problem of accurate die attach and should be avoided as far as possible. Du-Pont recently published a new recipe for transition vias for the mixed metal system, which is under investigation. Measurements show a significant reduction of the posting from around 20 to about 10µm using the new ink.



**Figure 21:** Posting effect: 10µm, new transition via compared to 20µm Conventional AgPd transition via

The progress beyond the state of the art is demonstrated by new packaging solutions recently developed at VIA for RF and non RF industrial applications and in the frame of research programmes.

### 17. 77 GHz Radar Front end

To introduce the new 9K7 LTCC material system into the manufacturing line to qualify the processes and to validate the system, it was decided to use the new system for the development of a very challenging 77 GHz Radar Front-end. This development was carried out in the frame of a joint cooperation research project, funded by the German BMWI/AiF.

The sensor developed aims at applications where the sensor is mounted very close - but contactless - to the target. The target can be measured by using the continuous wave (CW) mode of operation. Since the phase is ambiguous the measurement range is limited to half the wavelength. For example, in the case of a frequency of 77 GHz, these measurements range of  $\ddot{e}/2 = 1.95$  mm might be located at 1cm distance to the sensor with measurement accuracy in the µm range. The sensor's centre frequency is therefore a trade off between the measurement range, which decreases with frequency, and the precision, which increases. A very important point for the design is the available space for the sensor. The LTCC frontend takes only 1.4mm x 30mm x 12mm including a broadband Vivaldi antenna. The 9K7 LTCC process has been chosen for its RF capability, miniaturization capabilities and robustness in harsh environments. The key features of the Frontend, designed by IMST, are as the following:

Frequency	77 GHz (72 77 GHz)
Operation	CW and FMCW
Output Power	5 dBm
Antenna	Vivaldi
Module Size	30 x 12 x 1.4 mm3
Material	DuPont 9k7



**Figure 22:** VIA/IMST: 77 GHz Radar Front end, Substrate and assembled package

The following pictures are showing the radar module in different stages of the development:

# 18. Light weighted TR Module using CarfAl heat sinks

In the first run (compare Fig. 13) a conventional Molybdenum heat sink was bonded to the LTCC substrate, assembled and qualified.



Figure 23: TNO: TR-Module using CarfAl heat sink

Introducing the CarfAl material has lead to a weight saving of 80 % of the complete module!

## 19. High Speed Optical Router

A LTCC substrate has been used as an optical bench to integrate and optically interconnect a high speed data transmission chip. The accuracy challenges were extreme:

cavity with  $\pm$  50µm, blind holes  $\pm$  25µm, Vias 90µm diameter, Via position over the whole substrate  $\pm$  50µm, thickness 1,5mm 5 $\pm$  µm, Signal speed 10 Gbit/s, Flip chip pitch 100µm, BGA Pitch 500µm.



Figure 24: CEA: Optical Router, Schematic

A solution could be found by applying the 0-Shrinkage process, picosecond laser drilling, grinding and lapping the substrate and interconnecting with thinfilm on top and thickfilm on bottom to provide a full high accuracy solution.



Figure 25: Optical Router Package

## 20. One way DSC-Chip

A complete differential scanning calorimetric system was developed and integrated into a LTCC substrate. The key features have been:

- Integrated Temperature sensors
- Integrated heater elements
- Pinout in chip card format
- Total mass of the chip: 1,43g
- Measuring head: 400mg



Figure 26: One way DSC Chip

The system has been realised at VIA in Heraeus CT 700 and HL 800 material system.



**Figure 27:** DSC Chip in test fixture (University of Bayreuth)

## 21. Ceramic Microreaktor

BioMEMS and biosensors, Lab on chip systems and similar devices are in development and under investigation since a couple of years. One of the elements which VIA has developed is a highly integrated micro reactor, shown in the following figures.



Figure 28: LTCC Micro reactor

The micro reactor contains fully integrated fluidics and soldered fluidic interfaces. The key features are the 50 $\mu$ l reactor with a fluidic system allowing a flow rate of 1 $\mu$ l per minute.

Enzymes were immobilised onto a 0,1x0,4mm 3d channel.The cell growth in the reactor has been verified.

### Conclusions

LTCC is providing manifold advantages for packaging tasks in the field of high-end applications. Applying new materials like 9K7, new light weighted materials for packaging components like CarfAl, low cost materials for brazing like SnAg together with a proper man-



Figure 29: Micro reactor, schematic of the sensor and detail

agement of mixed metal processing; the LTCC technology will make a significant step ahead towards a cost efficient packaging technology. In addition with the well-known integration capabilities and its superior reliability performance it provides innovative and novel RF packaging solutions.

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# LTCC fluidic microsystems

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**Abstract:** In this paper, potential of the low temperature co-fired ceramics (LTCC) technology for fluidic microsystems fabrication is discussed. The authors give a brief overview of fluidic structures fabrication process in LTCC modules. The presented micro-fabrication techniques utilize sacrificial volume materials combined with multi-step isostatic lamination. Moreover, the design and construction of the few exemplary LTCC-based microfluidic systems developed at Faculty of Microsystem Electronics and Photonics at Wrocław University of Technology are presented.

Key words: Low temperature co-fired ceramics (LTCC), sacrificial volume material (SVM), microfluidic, microsystem

# Tekočinski LTCC mikrosistemi

**Povzetek:** V članku je opisan potencial keramičnih tehnologij z nizko temperaturo žganja (LTCC) za proizvodnjo tekočinskih mikrosistemov. Avtorji podajo kratek pregled proizvodnih procesov tekočih struktur v LTCC modulih. Predstavljene tehnike mikroproizvodnje uporabljajo žrtveni material v kombinaciji z večstopenjsko izostatično laminacijo. Poleg tega so predstavljene oblike in zgradbe primerov mikrotekočinskih sistemov na osnovi LTCC, ki so bili izdelani na Fakulteti za mikrosistemsko elektroniko in fotoniko na univerzi v Wroclawu.

Ključne besede: keramične tehnologije z nizko temperaturo žganja (LTCC), žrtveni material, mikro tekočina, mikro sistem

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# 1. Introduction

We can observe increased interest in the field of fluidic microsystems in the last two decades [1,2]. The fluidic microsystems are used in medicine, biology and analytical chemistry, as well in application in which fluid is used as a coolant. The main reason of this interest is rapid development of the microengineering techniques. Progress in microenginering enabled fabrication of miniature and precise fluidic structures (e.g. channels, cavities, chambers etc.) with characteristic dimensions from single millimeters to hundreds of nanometers. In a consequence it is possible to fabricate microsystems which are capable to handle with fluid in micro- or nanoliter volume range. Thanks to such significant reduction of the specimen volume the fluidic microsystems produce less wastes and are cheaper to use in comparison to classical laboratory apparatuses. Another aspect is the number of possible areas of the fluidic microsytstem applications: analytical chemistry, medical diagnosis, DNA sequencing, cell separation, microbiological analysis, high-throughput synthesis, environmental monitoring and others [3-6].

Nowadays fluidic microsystems are fabricated using silicon/glass [7], polymer [8], PCB (Printed Circuit Board) [9] and LTCC (Low Temperature Co-fired Ceramics) [10-12] technologies. The main advantages of the LTCC in comparison to silicon, polymers and PCB are chemical inactivity, chemical resistance and high temperature stability [13,14]. Moreover, the LTCC technology enables to accomplish both mechanical and electrical functions in a single ceramic module. The possibility of integration of fluidic structures, active and passive electronic components, optoelectronic devices, sensors, actuators, MEMS (micro electro-mechanical system) and package into one multilayer module is the main advantage of the LTCC over other mentioned technologies [15,16]. Moreover, the LTCC can be bonded with other materials using anodic bonding (LTCC-Si) [17], microwave plasma (LTCC-polymer) [18] or low temperature melting glass (LTCC-ceramic). Due to all above-mentioned advantages the LTCC can be a potential alternative for fluidic microsystems fabrication.

In this paper, the main aspects of the LTCC technology for fluidic microsystems fabrication are briefly discussed. Subsequently, an overview of the techniques for fabrication of precise 3D fluidic structures in the LTCC material using sacrificial volume materials (SVMs) is given. Finally, few exemplary LTCC-based fluidic microsystems are presented.

## 2. Fluidic structures fabrication

The section presents the SVM-based methods of threedimensional (3D) structuration of the LTCC module for the applications in fluidics. A typical LTCC module is built of several glass-ceramic tape layers, connecting vias, surface and buried conductors and passive components. The network of conductive lines and passives are deposited using screen-printing method. After printing various shapes (channels, cavities etc.) can be cut in green LTCC tapes using laser or milling machine. In the next step all LTCC tapes are stacked together and laminated. Typically the thermo-compression lamination process is performed at high pressure (up to 20 MPa), elevated temperature (up to 90 °C) for time of 5-30 minutes. After lamination the LTCC module is cofired according to two step thermal profile with a maximum temperature of 850-900 °C. The thermo-compressive lamination and co-firing processes provides very good bonding between individual LTCC tape layers, however, the conventional high pressure lamination methods pose some problems. High pressure and temperature of the process strongly affect the quality of the final fluidic structure and preclude realization of the complex 3D features such as: channels, cavities etc. In particular, the key challenge is the ability to form surface and buried fluidic structures without sagging during the technological process. Recently several techniques for fluidic structures fabrication have been discussed in the literature. The most common technologies for 3D processing of the LTCC tape are hot embossing [19], low pressure lamination methods [20,21] and techniques basing on sacrificial volume materials [22-24]. The SVM is a temporary insert which supports and defines fluidic structure during high pressure lamination process. Various types of materials are used as the SVMs e.g. wax, graphite, polymers, mineral materials [25,26]. Depending on the applied sacrificial volume material its elimination from LTCC module takes place either by dissociation during co-firing (polymers, graphite) or by etching or pouring out after firing (mineral materials). Commercial available SVMs are made of graphite because of its inherent features. It does not react with the LTCC material and can be easily applied as a paste or tape. Moreover, graphite burns away in air above 600 °C which is intermediate between debinding and sintering temperatures of the LTCC. The exemplary thermo-gravimetric curve of the graphite-based SVM paste is presented in Fig. 1.



**Figure 1:** TG analysis results of the graphite-based SVM paste.

Ideally a graphite-based sacrificial volume material should be completely removed below the temperature where the open porosity of the LTCC is closed by densification (about 800 °C). Therefore, it is recommended to modify co-firing profile by applying a slower ramp rate up to 850-900 °C or additional isothermal heating stage to assure complete graphite burnout. Unsuitable thermal profile may lead to swelling or contamination of the LTCC material.

There are two common techniques of the fluidic structures fabrication which are based on the applying of graphite-based SVM paste or tape. The LTCC-based fluidic structures can be fabricated using either "define and fill" or "collate and laminate" techniques. Fabrication of the fluidic structures with the feature size from 100 µm to single centimeters is possible.

#### 2.1. Define and fill

Scheme of the fluidic structure fabrication process in the LTCC using "define and fill" technique is presented in Fig. 2. In the first step of this technique a fluidic structure is cut in a green LTCC tape using laser system or milling machine. The cutting process is followed by thermo-compressive lamination of the middle and bottom LTCC tape layers. The first lamination is performed at relatively low pressure (about 1 MPa) and room temperature using isostatic or uniaxial press. After initial lamination the created fluidic structure is filled with a sacrificial volume material. The fluidic structure is filled in using screen-printing method. The SVM paste is printed through openings made in the backing polymer of the LTCC tape. The openings in the backing polymer are matched with openings in the middle LTCC tape layer. Application of the carrier film decreases number of the process steps because there is no need to make a stencil or specific pattern on the screen.



#### (b)

**Figure 2:** Fabrication of (a) open and (b) buried channel in LTCC module using "define and fill" technique.

Moreover, the SVM paste shrinks after drying and carrier film ensures proper thickness of dried SVM. The SVM deposition process is called "screeding" in the literature [25]. In the next step the fluidic structure is sealed with a top LTCC tape layer. Second lamination is performed at pressure of 10-20 MPa and at elevated temperature (40-70 °C) in a isostatic or uniaxial press. The LTCC laminate is co-fired in air with a modified thermal profile. The additional heating stage and temperature slow ramp rate assure complete burnout of the applied SVM. The "define and fill" technique can be used for fabrication of open and buried fluidic structures in the LTCC modules. Exemplary scanning electron microscope (SEM) image of the microchannel made in the LTCC using mentioned technique is presented in Fig. 3.



**Figure 3:** SEM image of the open channel made in the LTCC module using "define and fill" technique.

#### 2.2. Collate and laminate

Alternative method which is used for fluidic structures fabrication in the LTCC modules is called "collate and laminate". A flow-chart of this technique is presented in Fig. 4. The process starts from deposition of the pattern of the fluidic structure made of the SVM paste on green LTCC tape. In the next step the LTCC tape layer with deposited SVM is collated with another LTCC tape. The lamination process is performed at elevated pressure (5-20 MPa) and temperature (40-70 °C) using isostatic or uniaxial lamination press. In this technique there is no pre-existing fluidic structure. The fluidic structure is formed by pressure of the lamination process which deforms green LTCC material around the SVM paste and bonds compatible areas [27]. After lamination the LTCC module is co-fired with a maximum temperature of 850-900 °C. During co-firing process the SVM material is removed leaving empty volume. This technique enables to fabricate fluidic structures with characteristic dimension from 100 µm to few centimeters. SEM image of the exemplary fluidic structure made in LTCC using "collate and laminate" technique is presented in Fig. 5. The "collate and laminate" technique can also be used to fabricated tensile bars, conductor posts, membranes and suspended thick-film features [28].



**Figure 4:** Fabrication process of the fluidic structure using "collate and laminate" technique.



**Figure 5:** SEM image of the open channel made in the LTCC module using "collate and laminate" technique.

#### 3. LTCC-based microfluidic sytems

Modern microfluidic system is build of several functional blocks. These sub-systems are responsible for various functions: sample transport (micropumps and microvalves), preliminary preparation of the sample (micromixers), carrying out appropriate (bio)chemical reaction, product separation and detection. Each of sub-systems can work as separate device or can be integrated into one monolithic structure. All functional blocks can be fabricated using LTCC or hybrid LTCC-Si, LTCC-PDMS technologies.

#### 3.1. Microvalves

Microvalves are usually fabricated as a hybrid structures. They consist of moving part, usually flexible membrane, made of steel, silicon or polymer. Actuation of the membrane is provided by piezoelectric or electromagnetic principle. Construction of the initially open valve made in the LTCC is described in [29]. It consists of fluidic channels, cavity, valve seat and a steel membrane. The piezoelectric layer deposited on the steel membrane forms a unimorph piezoactuator. When electric field is applied transverse expansion and contraction of the piezoelectric layer occur. These deformation creates an internal bending moment and deflection of the structure. The unimorph piezoactuator generates approximately 1.3 µm displacement, which closes the valve. For valves based on electromagnetic principle vertical actuation is generated by interaction between magnetic field and a permanent magnet which is placed on flexible membrane. Gongora-Rubio et al. [30] presented a LTCC-based hybrid microvalve with electromagnetic actuation. It consists of fluidic channels and a silicon membrane with bonded permanent magnet. The fluidic channels and multilayer coil are made inside the LTCC module. Using SmCo magnet with 1 mm diameter it was possible to obtain 200 µm deflection of the Si membrane. Similar construction of the microvalve was developed at Faculty of Microsystem Electronics and Photonics at Wrocław University of Technology [31]. The valve was built of twelve LTCC tape layers and PDMS membrane with immersed permanent neodymium magnet. Scheme of the valve is presented in Fig. 6. Inlet and outlet fluidic channels and valve seat were cut in green LTCC tapes using Nd-YAG laser system. The PDMS membrane was bonded to fired LTCC structure using argon plasma. Fabricated valve is presented in Fig. 7. The presented valve is normally open. It can be closed by deflection of the PDMS membrane. The deflection is caused by magnetic force generated by current flowing through the multilayer coil.



**Figure 6:** Scheme of the PDMS/LTCC-based electromagnetic valve (not to scale).



**Figure 7:** PDMS/LTCC-based valve with electromagnetic actuation [31].

#### 3.2. Micromixers

The rapid mixing between two (or more) initially segregated fluids is often crucial to the effective functioning of a modern microfluidic system. Majority of (bio) chemical processes such as enzymatic reactions require intermix of all reagents for initiations. An efficient micromixer should mix very small volumes of fluids without taking much space in acceptable time-scales. However, it is very difficult to mix microvolumes of fluid, because the flow is in a laminar regime. As a consequence mixing process is based mainly on relatively slow molecular diffusion. Diffusive mixing time is given by equation (1):

$$t_{diff} \propto \frac{D_h^2}{D} \tag{1}$$

where  $D_h$  is a characteristic dimension of the mixer (m) and D is a coefficient of molecular diffusion (m<sup>2</sup>/s). The micromixers are classified as either active or passive. As can be noticed from equation (1) fluids which flow with a mean velocity of U have to pass through distance equal to  $U(D_h^2/D)$  to be completely mixed. Therefore, the mixing time and distance required to effective mixing can be very high. The magnitude of the mass flux of the fluid particles due to the molecular diffusion is described by equation (2):

$$J = -D\nabla_C \tag{2}$$

where c is particles concentration  $(m^{-3})$ . Using equation (2) it can be noticed that the key to efficient mixing relies mainly on amount of interface area between mixing fluids and ability to create high concentration gradients between fluids. Large interface area results in larger are for mass transfer and concentration gradients accelerate diffusion. Elongation of interface area and supporting concentration gradients can be obtained in microscale by stretching and folding phenomena which are characteristic for chaotic mixing. In order to provide effective mixing active and passive micromixers are used. In general, active micromixers require external energy (e.g. temperature, pressure, acoustics etc.) to disturb fluid flow pattern. Passive micromixers do not require external energy. Effective mixing is provided by micromixer's channel geometry. Spatial changes along the mixing channel axes result in frequent changes of fluid flow direction. The main advantage of the passive micromixers over active ones are easiness of fabrication and integration with other microfluidic devices. Active micromixer made with LTCC technology was presented by Bau and co-workers [32]. They shown design, realization and functioning of the magneto-hydro-dynamic (MHD) micromixer. The presented micromixer utilizes electro-magnetic (Lorentz) force to improve mixing phenomenon in microchannel. The Lorenz force is induced by coupling between magnetic and electric fields which are generated in the micromixer. The MHD mixer consisted of the LTCC structure and a permanent Neodymium magnet. Mixing channel with electrodes on the bottom was made inside the LTCC structure. Mixing channel was filled with electrolyte solution. When the electrical potential was applied to the pairs of electrode, currents were induced in the solution. Currents and magnetic force induced by permanent magnet generate the Lorenz force in a perpendicular direction to the magnetic and electric fields.



Figure 8: LTCC-based serpentine micromixer.

Direction of the force depends on positive and negative poles of a DC power supply. Rapid changes of the poles causes stretching and folding of the electrolyte solution in the mixing channel. The high efficient passive micromixer was developed at Faculty of Microsystem Electronis and Photonics at Wrocław University of Technology [33]. The micromixer was composed of sequence of bends arranged in I-shape serpentine. The serpentine micromixer is presented in Fig. 8.



**Figure 9:** Concentration distribution in a serpentine passive micromixer for (a) low and (b) high flow rates.

The efficiency of the presented micromixer relies on inertial effects. For relatively low flow rates the mixing is poor, but for higher flow rates the mixing efficiency increases. Results of mixing modeling for low and high flow rates are shown in Fig. 9. According to numerical simulations for low flow rates viscous effects dominate, therefore, bend-induced fluid recirculation and flow pattern disturbances decay rapidly. However, for higher flow rates the mixing process is more efficient. As can be seen from Fig. 9b both fluids remain separated only in the vicinity of the junction and are well mixed after passing few bends of the serpentine. For relatively high flow rates the interface area between mixing fluids enlarges due to the stretch and fold phenomena. Large interface area between fluids creates more space for the fluid particles for diffusion and enhances mixing process.

#### 3.3. Microreactors

Microreactor technology has become very promising in the fields of chemistry, biotechnology and process enginnering. A microreactor is a miniature device where appropriate (bio)chemical reaction occurs. The microreactor needs very small amounts of reagents for operation. The microreactors work either as a standalone devices or as a part of more sophisticated analytical system. The stand-alone microreactors are mainly used for evaluation of the influence of different chemical compounds and drugs on enzyme activity and high throughput chemical synthesis. In general, they can be classified as a batch type or flow-through microreactors. The batch type microreactors are filled with a catalytic bed made of porous material. The catalyst (e.g. enzyme) is immobilized on the surface of the porous carrier. For flow-through microreactors the catalyst is immobilized on its channels walls. Exemplary batch type microreactor made of LTCC is presented in Fig. 10. It consists of two chambers separated with a threshold and integrated heater and temperature sensor. Heater provides uniform temperature distribution in the whole area of the reaction chamber. The catalytic bed in the form of porous glass or polymeric beads with immobilized enzyme (urease) is placed in a larger chamber of the microreactor. The threshold precludes catalytic bed to move to the chamber for reaction products. A LTCC-based flow-through microreactor is presented in Fig. 11. It is composed of inlet and outlet chambers connected with sixteen parallel microchannels and integrated heater. Enzyme is immobilized on the surface of the microchannel walls.

The presented microreactors were used for urea determination in biological fluids [34]. The principle of their operation is based on hydrolysis of urea catalyzed by urease. One of the reaction products are hydroxyl ions which are used for indirect determination of urea in the sample.

#### 3.4. Detection unit

Detector is one of the most important part of the substantial number of fluidic systems. It is responsible for qualitative or quantitative detection of the analyte in the liquid sample. A very small volumes of the fluid can be analyzed using either electrochemical or optical methods. The optical methods are characterized by very good sensitivity and repeatability. The LTCC-based optical detection module for absorbance measurement is presented in Fig. 12.



**Figure 10:** Batch type microreactor made with LTCC technology.



**Figure 11:** Flow-through microreactor made with LTCC technology.

It consists of U-shaped microfluidic channel and two polymeric optical fibers [35]. Space between optical fibers creates 10 mm long absorption cell. The input optical fiber is connected with light emitting diode and the output optical fiber is connected to light-tovoltage converter. Principle of operation is based on selective light absorption by the analyte. A green light  $(\lambda_{max} = 565 \text{ nm})$  from LED is transmitted via input optical fiber to the absorption cell. The analyte which flows between two optical fibers absorbs a portion of light. The rest of the light is transmitted via output optical fiber to the light-to-voltage converter. The changes of the light intensity on the light-to-voltage converter are proportional to the changes of the analyte concentrations. According to the Beer's law magnitude of the absorbed light A is proportional to concentration of the analyte:





**Figure 12:** LTCC-based detection unit for absorbance measurements: (a) schematic view and (b) photograph.

$$A \equiv \log \frac{I_0}{I} = cel \tag{3}$$

where  $I_o$  and I are intensities of the light before and after absorption, c is the molar concentration of the analyte (M),  $\varepsilon$  is the molar absorptivity (cm<sup>-1</sup> M<sup>-1</sup>) and I is the absorption cell length (cm). It can be seen that for fixed length of absorption cell and the same molar absorptivity of the analyte the absorbance is proportional to the concentration of the analyte. Utilizing the light-to-voltage converter as a light detector it is possible to measure the optical absorbance using following formula:

$$A \equiv \log \frac{I_0}{I(c)} = \log \frac{U_o}{U(c)} \tag{4}$$

where  $U_o$  is the output voltage for distilled water and U(c) is the output voltage for analyte with concentration c. Exemplary dynamic response of the presented LTCC detection module for various concentrations of potassium permanganate (KMnO<sub>4</sub>) is presented in Fig. 13. As can be seen from Fig. 13 the presented LTCC detection module is characterized by high signal-to-noise ratio and good repeatability of the output signal.



**Figure 13:** Dynamic response of the LTCC detection module for various concentrations of  $KMnO_4$  test solution ( $\lambda_{max} = 565 \text{ nm}$ )

# 4. Conclusions

Short overview of the fluidic structures fabrication methods in LTCC has been discussed in the paper. The presented methods are based on application of the sacrificial volume materials. Using SVM it is possible to fabricate various fluidic structures with a characteristic dimensions from hundreds microns to single centimeters. By applying appropriate technological procedure it is possible to fabricate open or buried fluidic structures in the LTCC multilayer module.

The exemplary LTCC-based fluidic microsystems (valve, micromixer, microreactor and optical detection module) developed and fabricated at Faculty of Microsystem Electronics and Photonics at Wrocław University of Technology has been presented. The presented fluidic microsystems has been fabricated using SVM-based fabrication methods.

The performed research has shown the LTCC technology potential to fabricate fluidic microsystems. The presented technology can be successfully applied for fabrication of all functional blocks of the integrated micro-total analysis systems or lab-on-chip devices.

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# 3D structuration of LTCC and related technologies for thermal management and microfluidic structures

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**Abstract:** Ceramic technologies such as LTCC (Low Temperature Co-fired Ceramic) and thick-film are used widely in electronic circuits exposed to harsh environments, for applications in fields such as aerospace, automotive and energy exploration, where, owing to their thermal and chemical stability, they have an extensive and successful track record. Recently, the extensive structuration possibilities afforded by LTCC have led to its use in sensors, microfluidics and thermal management (hotplates). In the first part of this work, we present both new and classical techniques for structuring ceramic devices for thermal management, microfluidics or both. Critical aspects for achieving successful structuration and reliable device operation are discussed, such as lamination and sealing techniques, materials formulation and selection, as well as thermomechanical design. These considerations are illustrated in the second part of this work with several examples: micro-hotplates for various applications, microfluidic coolers, chemical reactors and solid-oxide fuel cell (SOFC) components.

Key words: Thick-film technology, LTCC, 3D structuration, microfluidics, thermal management.

# 3D strukturiranje LTCC in sorodnih tehnologij za termično upravljanje in mikro tekočinske strukture

**Povzetek:** Keramične tehnologije, kot je LTCC (keramika z nizko temperaturo žganja) in debeli sloji, se pogosto uporabljajo v elektronskih vezjih, ki so izpostavljena neugodnemu okolju, za aplikacije v vesolju, avtomobilskih in energetskih raziskavah, kjer imajo s svojo termično in kemijsko stabilnostjo uspešno in dolgo zgodovino. Velike možnosti strukturiranja, ki jih nudi LTCC so omogočile njihovo uporabo v senzorjih, mikro tekočinah in termičnem upravljanju (hotplate – vroča plošča). V prvem delu članka predstavljamo klasično metodo strukturiranja keramičnih elementov za termično upravljanje, za mikro tekočine ali oboje. Opisani so kritični vidiki za doseganje uspešnega strukturiranja in zanesljivega delovanja, kot je laminacija, tehnike pečatenja, formuliranja in izbora materialov, kakor tudi termomehanična oblika. Te odločitve so v drugem delu članka predstavljene s pomočjo naslednjih primerov: mikro vroča plošča za različne aplikacije, mikro tekočinski hladilniki, kemični reaktorji in deli gorivnih celic s trdnim elektrolitom (SOFC).

Ključne besede: debeloplastna tehnologija, LTCC, 3D strukturiranost, mikro tekočine, termično upravljanje.

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# 1. Introduction

Originally introduced as a chip / multichip module packaging and high-reliability circuit technology [1-4], LTCC has found important additional applications in the field of advanced packaging, sensors and microfluidics. This requires more advanced 3D structuration techniques, required to form features such as thin bridges, cavities, membranes, channels and hotplates [5-16]. In the green state, LTCC tape is easily shapeable, and may be cut and further processed by a wide variety of methods (Table 1), which in principle easily allows features such as channels, bridges and membranes (Figure 1). An overview of the resulting LTCC applications is given in Table 2 (see also other paper at this conference [17] for mechanical sensors). The very wide range of devices and applications attest for the excellent 3D structurability of LTCC. However, problems that appear at the different stages of processing (handling, lamination and firing, Table 1) in practice severely hamper many applications. Moreover, the properties and limitations of LTCC as a material must also be taken into account, such as mechanical strength (short- and long-term), chemical durability and thermal stability. Also, physical properties such as the coefficient of thermal expansion (CTE) and elastic modulus are important for device performance.

The purpose of the present work is therefore to give an overview of the applications of LTCC structuration, centred on microfluidics and thermal management, with the associated themes:

- Processing issues and how they are resolved
- Properties and limitations of LTCC as a material
- Implications on device design



**Figure 1:** Example LTCC structures (see table 2) (1-3) Meander channel (1) and heater module (2) for gas viscosity sensor (3, with membrane) [18-20]; 4) chemical microreactor with complex fluidic circuit [16, 20]; 5) thermal bubble inclinometer (with membrane) [9]; 6) cantilever sensor for low forces [9, 16, 21, 22].

**Table 1:** Methods and operations used in 3D structuration of LTCC.† SVM = sacrificial volume material. # MSM / FSM = mineral / fugitive sacrificial (volume) material.

Operation	Methods
Cutting / drilling / shaping	<ul> <li>Mechanical microdrilling / end milling [7, 23]</li> <li>Punching / stamping [24]</li> <li>Laser cutting of LTCC [24-26]; of conductors [27, 28]</li> <li>Embossing [24, 29, 30]</li> <li>Controlled laser ablation [31]</li> <li>Solvent vapour jet cutting [32]</li> <li>None (lamination directly around SVM†) [7, 33, 34]</li> </ul>

Lamination methods and conditions (LP/HP = low-/high- pressure)	<ul> <li>No sacrificial material</li> <li>Uniaxial, HP, cold [13, 35]</li> <li>Uniaxial, minimal-pressure, cold [36] or warm [37]</li> <li>Adhesive tape, LP [38, 39]</li> <li>Solvent / adhesive paste / adhesive solution, LP [12, 40-44]</li> <li>Hot-melt adhesive layer, LP [45]</li> </ul>
	With sacrificial material - Warm, HP, uniaxial or isostatic (standard methods)
Lamination order	<ul> <li>All at once (standard procedure)</li> <li>Sequence of partial laminations, often with different methods/ parameters [13, 44, 46, 47]</li> </ul>
Firing	No sacrificial material or MSM# - Standard, in air (usually)
	<ul> <li>With FSM#</li> <li>Air (match sintering and burnout kinetics) [10, 48-51]</li> <li>Air-N2-air (sinter in N2, then oxidise FSM) [17, 52]</li> </ul>
Post-firing operations (depending on device)	<ul> <li>MSM† removal by chemical dissolution [52-57] or mechanical blowing [8]</li> <li>Screen-printing of materials incompatible with co-firing</li> <li>Cutting of temporary supports [58 2012]</li> <li>Singulation by dicing or breaking</li> </ul>

**Table 2:** Applications of LTCC structuration techniques beyond purely electrical ones. + M(O)EMS: micro (opto) electromechanical system.  $\# \mu$ -SOFC: micro solid-oxide fuel cells.

Field	Applications
Advanced & high- reliability hermetic packaging	<ul> <li>MOEMS† package [59]</li> <li>Package + quality control [60, 61]</li> <li>MEMS pressure sensor package for medical applications [62]</li> <li>Active getter module [63]</li> </ul>
Pressure sensing	<ul> <li>Piezoresistive (membrane) [64, 65]</li> <li>Piezoresistive high-pressure cell (direct compression) [20, 66]</li> <li>Piezoelectric (resonance) [67]</li> <li>Capacitive (membrane) [68, 69]</li> <li>Complete piezoresistive pressure sensor (+electronics) [15, 17, 70, 71]</li> </ul>
Force & accel. sensing	<ul> <li>Low forces [9, 16, 21, 22]; applied to low pressures (indirect) [72]</li> <li>Acceleration [17]</li> </ul>
Optical sensors	<ul><li>pH [73]</li><li>Absorbance [74] &amp; fluorescence [75]</li></ul>
Flow & liquid sensing	<ul> <li>Fuel injection (thermal) [76]</li> <li>Flow sensor, thermal [17, 71, 77, 78] or mechanical [79]</li> <li>Thermal bubble inclinometer [9]</li> </ul>
Flow con- trol	<ul><li>Valve [80]</li><li>Substrate for electrovalves [13]</li></ul>

Liquid mi- cro- reactors	<ul> <li>Emulsifier [81], dilution device [82]</li> <li>Mixer [83, 84]</li> <li>Particle synthesis [85]</li> <li>Electrochemical [86]</li> <li>Polymerase chain reaction [87]</li> <li>Photocatalysis [88]</li> <li>Integrated, + flow sensing &amp; calorimetry [11, 20, 89] / flow &amp; pressure &amp; liquid sensing [90]</li> </ul>
Gas micro- reactors	<ul> <li>H<sub>2</sub>-O<sub>2</sub> combustor [91]</li> <li>Gas burner with window [41]</li> <li>Fuel cell reformer [92, 93]</li> <li>Micro-plasma generator [94]</li> </ul>
Hotplates	<ul> <li>For gas sensing [27, 95-97]</li> <li>Scanning microcalorimeter [98]</li> <li>Atomic clock module [99, 100]</li> <li>Fluidic &amp; thermal package for MEMS† μ-SOFC# [101, 102] and μ-thrusters [103]</li> </ul>
Multi- sensors /-physics	<ul> <li>Flow / pressure / temperature sensor for compressed air [46, 104]</li> <li>Gas viscosity &amp; thermal conductivity sensor [18-20]</li> </ul>
Micro- thrusters	<ul> <li>Liquid fuel [44, 105, 106]</li> <li>Solid propellant [107]</li> </ul>

# 2. Processing techniques

The present section reviews the different techniques and issues at each stage of processing. Processing issues are usually exacerbated when using fine structures, such as required for sensitive device.

#### 2.1. Basic processing routes

The most straightforward processing route is the "cutand-laminate" one, whereby each LTCC laser is simply cut (usually by laser), with the resulting stack then being uniaxially laminated and fired (Figure 2). This simple route is feasible for applications involving relatively robust structures, where crushing and sagging of layers are not a big issue, such as the chemical microreactor shown on Figures 5 & 6.



**Figure 2:** "Cut-and-laminate" structuration of LTCC: uniaxial lamination of previously processed sheets between rigid metal plates.

If slender membranes or bridges are used, sacrificial volume materials (SVMs) may be used to avoid crushing / sagging (Figure 3), using uniaxial or (pseudo-)isostatic lamination.



**Figure 3:** Filling crushable cavities with SVM, with pseudo-isostatic lamination (isostatic or uniaxial also possible).

Finally, cavities may be directly created by printing SVM onto LTCC, without removing the corresponding volume from the tape (Figure 4), which requires (pseudo-) isostatic lamination.



**Figure 4:** Pseudo-isostatic (similar to isostatic, where the bottom face is also deformed) lamination of LTCC modules, with printed SVM to create cavities.

#### 2.2. Handling during screen-printing

Structural features such as intricate channels and thin bridges exacerbate the usual difficulties of handling fine LTCC tapes. Especially, simply cutting out complex channel networks (Figure 6) in one layer is difficult or even impossible, as this would structurally separate the tape, or weaken it excessively. To get around this issue, two techniques are commonly used:

"Stitching" the fluidic circuit across several layers allows fabrication of complex and strongly meandering structures, as often seen in fluidic process devices such as microreactors (Table 2, corresponding section; Figure 6), using the classical "cut-and-laminate" route. This requires an additional layer (or two, if crossovers are desired),

and creates some additional dead volumes due to alignment tolerances.

Sacrificial volume materials (SVMs) allow "printing" of channels without requiring the tape to be cut out: the channel is then formed during or after firing by removal of the SVM (Figure 4). However, this technique does have restrictions: it requires "standard", high-pressure isostatic or pseudoisostatic lamination to deform the LTCC tapes around the printed SVM, and results in significant deformation of the tapes and of the surface of the device [8, 11, 13, 59], which may not be the most convenient method for shaping other structures such as large cavities. Also, there are practical limits to the achievable aspect ratios, stemming from the screen printing process and restricted deformability of the LTCC tapes [14]. Finally, printing large amounts of sacrificial material can destroy the tape through attack from the SVM paste solvent. However, recent developments in screen-printing vehicle formulation show progress in formulating SVM inks that have low aggressivity towards LTCC tapes, and even allow removal of misprints by rinsing in water [33, 34, 108].





**Figure 5:** Chemical microreactor: device (top), LTCC module with superposed fluidic layout (middle) and complete electrical & fluidic layout (bottom). 12) Inlets & heat-up meanders; 3) outlet; 4) flow sensors; 5) bottom alumina heat spreader, below LTCC; 6) alumina heat shield for reaction zone. A) Thermistor for body temperature; B) thermistor for reaction zone temperature; C) resistor for heat output calibration.

#### 2.3. Lamination and firing

Optimal lamination is often a compromise: applying excessive pressure and temperature can result in deformation and crushing of cavities, while the reverse yields poor bonding (Figure 7). Several techniques, described in section 3, have therefore been developed to alleviate this issue in difficult cases: SVMs to protect cavities, and "glues" to facilitate lamination.



**Figure 6:** Chemical microreactor: LTCC layers, in unfired state, showing cut-outs for fluidic channels and thermal decoupling of calorimetric reaction zone. 1) Bottom wall; 2) bottom fluidic layer; 3) fluidic separation layer; 4) top fluidic layer; 5) top lid.



**Figure 7:** Lamination problems in fluidic structures: A) crushing of cavities; B) deformations combined with poor interlayer lamination below cavity due to absence of pressure; C) poor lamination, at arrow; B) good lamination with low deformation, with correct parameters [13].

Firing of slender structures also may lead to deformations, stemming from shrinkage mismatch between LTCC, functional materials and SVM, or simply from sagging of structures under their own weight. This may again be counteracted by SVMs, which however must be correctly formulated to avoid imparting deformations themselves during LTCC sintering [10, 13, 46, 52, 57].

## 3. SVM & Adhesive formulations

In addition to the LTCC tape itself and the assorted functional materials (conductors, resistors, etc.), 3D structuration may use two types of "auxiliary" materials, which are eventually removed during processing: 1) SVMs and 2) adhesives for low-pressure lamination.

#### 3.1. Sacrificial volume materials (SVMs)

SVMs support the 3D LTCC structure during lamination, avoiding crushing, or may even be used to create cavities by themselves (see 2.1 / Figure 4). If just used to fill existing cavities, they tend to add complexity to the manufacturing process, but careful design, e.g. limiting SVM deposition to just one layer, or special processes such as filling with liquid wax [43], reduce this inconvenient.

A wide variety of SVMs have been investigated (Table 3), with carbon-based pastes or tape inserts being by far the most common.

Carbon / wax / polymer-based compositions are labelled fugitive sacrificial materials (FSMs), as they escape during firing, by evaporation, pyrolysis or oxidation to CO/CO<sub>2</sub>, which may lead to sagging during sintering through loss of support. To avoid this issue, carbon-based (mostly graphite) materials may be used; graphite is "semi-fugitive", as it is stable to very high temperatures in inert atmospheres, and only begins to oxidise rapidly above 600-650°C in oxygen-containing ones. This allows two strategies (see Table 1, firing):

- **1 Firing in air**, carefully matching sintering of LTCC with graphite oxidation kinetics by varying temperature rise rate and graphite particle size [8, 10, 48-51]
- 2 Sintering in inert atmosphere: burnout in air, up to ca. 600°C, followed by sintering in nitrogen (which preserves the graphite), and final oxidation of the graphite by switching back to air [17, 52].

Firing in air may be carried out also without restrictions, using mineral sacrificial materials (MSMs), which however requires an additional post-firing chemical or mechanical removal step (Table 1). This restricts in practice MSMs to open structures such as cantilevers or bridges on the surface of substrates [53-57, 109]. Further issues lie shrinkage mismatch, chemical interactions and limited chemical stability of some fired LTCC materials [57]. **Table 3:** Parameters and their values. † Applied to classical thick-film technology on Al<sub>2</sub>O<sub>3</sub>.

Туре	Sacrificial volume material (SVM)
FSM (fugitive)	<ul> <li>Carbon paste (printed) [10, 33, 34, 49-52, 108] or tape insert [8, 43]</li> <li>Wax, screen-printed [7] or filled as liquid [43]</li> <li>Kapton foil, laser-ablated [7]</li> </ul>
MSM (mineral)	<ul> <li>Al<sub>2</sub>O<sub>3</sub> setter tape [8]</li> <li>PbO-2SiO<sub>2</sub> glass [52]</li> <li>CaO-B<sub>2</sub>O<sub>3</sub> [109]; CaO-borax [53]</li> <li>Au [110]</li> <li>CaCO<sub>3</sub> + C [55]</li> <li>MgO-CaB<sub>2</sub>O<sub>4</sub> [57]</li> <li>SrCO<sub>3</sub> + [54]</li> <li>MgO-B<sub>2</sub>O<sub>4</sub> [56]</li> </ul>

#### 3.2. Lamination adhesives

In many cases, deformations mainly stem from the high pressures required to achieve good lamination. Moreover, simple uniaxial lamination of multilaver structures intrinsically faces the issues of low stresses above cavities (Figure 7B). Therefore, many techniques have been investigated to achieve satisfactory lamination guality at moderate pressures and temperatures (see Table 1, lamination), the most common being 1) application of adhesive tapes, and 2) printing of liquid / paste adhesives or solvents. There are however some drawbacks to these methods, as they require careful application of the adhesive, and handling of the resulting sticky LTCC tape can be quite cumbersome. Therefore, in order to facilitate handling, we recently proposed an alternative method using hot-melt adhesive layers [45], which are first generically deposited onto the LTCC tape. The adhesive is formulated to be tack-free or low-tack in ambient conditions, facilitating handling and minimising dust pickup, and then melt at moderate (≤60°C) temperatures, allowing low-pressure lamination at moderate temperatures.

During lamination, adhesives interact with the tape, and assist binding at low temperatures. The additional amount of organic material must be accounted for by somewhat lengthening the debinding step.

# 4. Materials limitations of LTCC

Fired LTCC material properties are typical of glass-ceramic materials (brittleness, relatively good thermal stability), and may be compared to thick-film multilayer dielectrics, from which they are derived.

#### 4.1. Mechanical strength

LTCC has somewhat lower short-term strength than alumina, of the order of 300 MPa, depending on the grade [111-114]. In sensitive mechanical structures such as low-range force and pressure sensors, this is offset by a much lower elastic modulus [113, 115], of the order of 100 GPa, yielding a comparable strain, i.e. resulting signal.

However, ceramics may be susceptible to stress corrosion in the presence of humidity, which must be accounted for in device design. This ageing behaviour is more severe in glassy ceramics such as LTCC than in standard 96% thick-film grade alumina, with glass-free materials such as yttria-stabilised zirconia (YSZ) and zirconia-toughened alumina (ZTA) essentially unaffected at ambient temperatures. To complicate matters, short-and especially long-term strength is affected by overlying thick-film materials, an effect that has yet to be studied on LTCC [112, 116, 117].

#### 4.2. Chemical durability

Whereas fired LTCC may be expected to be resistant to organic solvents, chemical durability in aqueous environments shows very strong variations: on the one hand, some materials (such as Du Pont 951) allow short-term operation ( $\approx$ 1 day) of microreactors with aggressive chemicals such as HCl and NaOH at concentrations >1 M. On the other hand, 3D structuration of LTCC using MSM was found to be hindered by degradation of LTCC in the relatively weak acetic acid used to dissolve the MSM [57]. Other studies also yielded very contrasting results, depending the LTCC material [118-120].

#### 4.3. Thermal stability & expansion

Essentially all common LTCC grades exhibit reasonable thermal stability up to ca. 500°C. Above this temperature, performance depends on the phase assemblage and chemical composition, with the more crystalline, essentially alkali-free materials exhibiting good mechanical stability and high resistivity at temperatures in excess of 600°C [115, 121]. This, together with the moderate thermal conductivity and CTE [113-115], allows creation of a wide range of hotplate structures (Table 2).

## 5. Conclusions

Due to its advantageous properties and relative ease of 3D structuration, LTCC has recently found wide appli-

cation in fluidic and/or heater structures. This trend is expected to intensify, due by advances in process technology and materials characterisation.

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### Cofired Platinum /Alumina Microsystems for Implantable Medical Applications

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**Abstract:** With the desire for reduction in size and increased requirements for higher input/output (I/O) for implantable microsystems, cofired platinum/ high temperature alumina cofire ceramic (HTCC) is emerging as a technology of choice due to the long reliable history of Pt wire /alumina ceramics use as feedthrough structures in pacemakers. The new developments require the development of brazing processes to attach a titanium seal ring to develop a hermetic enclosure and feedthrough requirements into the hundreds of I/Os for neurostimulator applications. This work evaluates the development of a cofired Pt/Alumina materials system, including interconnects and high density via structures. Reactions previously unreported have been investigated, including the catalytic reaction with organic binders, the reduction of alumina with platinum into PtAI3 and the reduction of the melting point of platinum 3000 C below its melting point, independent of the particle size (nano to micron size particles) or particle morphology. Firing atmosphere (air, hydrogen, inert), firing profiles and additives to control thermal expansion was evaluated to minimize exothermic reaction using high temperature X-ray diffraction, FIB nano-machining, SEM with EDS analysis and TEM for each of these conditions.

Key words: High density feedthrough, HTCC, cofired platinum- alumina

### Žgani mikrosistemi platina/aluminijev oksid za implantacijske medicinske naprave

**Povzetek:** Z zahtevami po zmanjševanju velikosti in povečevanju vhodno/izhodnih (I/O) zahtev implantacijskih naprav se je, zaradi že dolgo uporabljene keramike z žico iz platine / aluminijevim oksidom v vzpodbujevalcih srca, pojavila možnost uporabe žgane platine / žgane keramike z visokotemperaturnim aluminijevim oksidom. Nov razvoj zahteva uporabo procesa spajkanja titanovega tesnilnega obroča za izdelavo hermetičnega sistema s številnimi I/O prehodi za uporabo v nevrostimulatorjih. Delo ovrednoti razvoj žganih Pt/aluminijev oksid materialov skupaj s povezavami in veznimi strukturami visoke gostote. Še neobjavljene reakcije so raziskane skupaj s katalitično reakcijo organskih vezi, redukcijo Al oksida s platino v PtAl3 in znižanjem točke taljenja platine 300°C pod njegovo točko talitve neodvisno od velikosti delcev (nano do mikro velikosti) ali morfologije delcev. Atmosfera žganja (zrak, vodik, inertni plin), profil žganja in aditivi za kontroliranje termičnega raztezanja so obravnavani v smislu minimiziranja eksotermične reakcije z uporabo v visokotemperaturne difrakcije x-žarkov, FIB nano strojno obdelavo, SEM skupaj z EDS analizo in TEM za vsako od opisanih okoliščin.

Ključne besede: pretok visoke gostote, HTCC, žgana platina/aluminijev oksid

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### 1. Introduction

Platinum metallization is finding increases applications in implantable systems, especially in neural stimulator. Platinum has a long history as a conductor wire feedthrough materials for low input/output devices like pacemakers and implantable defibrillators or as feedthroughs and leads [1, 2] in higher output neural stimulator devices such as implantable cochlea, retinal implants and deep brain and muscle stimulators. Platinum is readily weldable and solderable, leading to easily second level assembly processes for microelectronic modules within the hermetic enclosure or attachment of micro lead structures. With the development of medical devices such as the implantable retina, the possibility of having I/O structures in the 300 to 1000 range is becoming a possibility. Additionally, neurological implants often require a much smaller device configuration footprint to minimize physiological interaction and external, RF coupled power coupling configurations to minimize the size of the device. As an example, the housing for one implantable retina under development is small enough to be attached to the eye to allow the microelectronics assembly to rotate with eye movement. The combined demands of small size, high I/O count, hermeticity and biocompatibility has pushed the development of a new class of cofired platinum / alumina feedthrough, leads [3] and microcircuit structures with I/Os in excess of 40/mm<sup>2</sup>, gold brazed to a titanium housing which is welded to provide a hermetic enclosure capable of meeting the hermeticity requirement of 1 X 10<sup>-9</sup> cc He/sec/atm , currently found in FDA approved medical devices [4, 5].

The bonding mechanism between platinum and alumina is believed to be by solid-state ceramic-metal diffusion bonding. However, despite different reaction mechanisms and products that have already been reported for the  $Pt/Al_2O_3$  system, the exact bonding mechanism between them is not clear [6-9].

The glass phase of the alumina-glass ceramic plays several important roles in ceramic/metal bonding. It anchors the metal to the ceramic by a combination of mechanical interlocking and chemical bonding and ensuring the hermeticity by filling the porosity in the metal [10]. During sintering of the substrate, a silica based liquid is formed between the alumina grains and penetrates the porous, fine-grained metallization layers. This occurs because of the capillary pressure difference between the porous alumina layer and the porous metal layer due to the smaller grain size of the latter and difference between their surface tension [11]. The contact angle of glass/ceramic is almost constant over the range of temperature and is about 30°. However, with refrectory metals such as Mo and W, the contact angle of the glass/ metal interface decreases with increasing firing temperature in a hydrogen atmosphere [10-12]. The contact angle of glass/platinum does not change uniformly with temperature. It decreases with increasing the temperature up to about 1150° C and then increases. This change of behavior can be due to the change of surface properties of platinum with temperature [13]. This change of behavior can change the effect of the glass in metal/ ceramic binding.

Recently, Lu et al. [14] and Suppel et al. [15] showed that the strength of direct bonding  $Pt/Al_2O_3$  depends basically on the crystal orientation of platinum and alumina. When the orientation is suitable for semi-coherency, the interface has a strong driving force to adopt direct metal-ceramic bonding; when the orientation is not suitable for coherency, then a weaker metal-glass-ceramic bond is favored since the covalent bonds in the glass and alumina are more conducive to mutual bonding.

In the current paper, the effect of glass migration mechanism and effect of the via diameter are studied initially, then the evaluation of the pt/alumina reation and effect of temperature on the microstructure and diffusion processes.

### 2. Experiments

Conventional high temperature cofired ceramics (HTCC) processing was used to develop the feedthrough [16]. 92% ceramic alumina tape (AdTech Ceramics Co.) and two different platinum powder, a spray dried and sintered Pt black platinum (Heraeus Inc. PM-100-10) and a spherical Pt (2  $\mu$ m) from Alfa Asar were used as the primary materials. The inks were mixed on a Hoover Color Muller parallel plate mixer. The ceramic tape was punched with a Keko PAM mechanical punch machine and punched vias were filled with platinum ink by the means of a PTC vacuum assisted bladder filler. Filled tapes were stacked, isostatically laminated and fired up to the temperature range of 1050-1550°C in different atmosphere.

Shrinkage of platinum powder and ceramic alumina tape was measured by the Horizontal optical dilatometer Misura® ODLT. TA Q600 SDT machine was used to measure the colorimetric behavior of the platinum in high temperature. JEOL JSM-6330F FE/SEM was used to capture the secondary electron image of the samples. Siemens x-ray system with MoKa ( $\lambda = 0.71073$ Å) radiation was used to measure the crystal structure of the samples. High temperature *in-situ* X-ray diffraction experimnts were conducted on the beam line B-2 ( $\lambda = 0.485946$ Å) of the Cornell High Energy Synchrotron Source (CHESS). Diffracted x-rays were collected between Bragg angles of  $2\theta$ =5° and  $2\theta$ =25° using a MAR3450 imaging detector.

### 3. Results and Discussion

### 3.1. Effect of Heating Rate

Figure 1 showed the densification behavior of nano platinum and alumina tape with temperature with the heating rate of 5°C/min up to the temperature 1500 °C.

As it can be seen in about 1500 C the density of nano platinum increased about 35%, while alumina's was increased only about 17%. The high difference in densification and sintering kinetics of platinum and alumina could create defect like cracks and camber in the final multilayer assembly. However, densification behavior of the platinum and ceramic could be controlled by the heating rate. Basically higher densification can achieve by lowering the heating rate [17].



**Figure 1:** The nonisothermal densification behavior of platinum powder and alumina tape.

SEM images of feedthrough assembly fired in four different heating rates are showed in figure 2 and a summary of their densification results are also showed in table 1. Each measurement in the table 1 is the average of the 5 – 10 platinum via in one sample.



**Figure 2:** Backscatter SEM image of the platinum via fired in different heating rate. a) 0.2 °C/min, b) 1 °C/min, c) 5 °C/min and d) 20 °C/min.

As it can be seen in the pictures all of the four samples showed delamination of metal from ceramic and camber generation. Still, the results in table 1 showed that the minimum difference between platinum and alumina archived with the heating rate around 5 °C/min.

**Table 1:** Densification behavior of platinum and alu-mina in different heating rate.

Heating rate (°C/min)	Platinum (µm)	Alumina (µm)	Difference (µm)
0.2	64.068	86.891	22.822
1	66.287	87.707	21.42
5	68.36	82.37	14.01
20	69.775	88.043	18.268

Diameter of platinum via metallization is decreased by decreasing the heating, while we cannot see such behavior for alumina. Decreasing the heating rate increases the shrinkage of the alumina. On the other hand the interaction of platinum and alumina can increase the adhesion force between them. The strong metal support interaction that was observed several times in the platinum supported catalyst, could describe this interaction. Hwang et. al. [18] and Luo et. al. [19] showed that platinum aluminate (i.e. Pt\_Al\_O\_) structure could be formed in the process of sintering Pt/Alumina mixture; in addition to that low heating rate can increase the diffusion of platinum to alumina and alumina to platinum and increase their physical adhesion. In result of such interaction, platinum tries to pull in alumina with itself. However the interaction force is not enough, which makes ceramic to tear apart in the interface area as it can be seen in the figure 2.

It should be noted, that the hole which can be seen in almost all of the samples could be attributed to the formation of volatile platinum oxide which is usually formed in temperatures above 650 °C [20].

### 3.2. Glass migration beahviour

As shown previously, camber developed in the via and the platinum delaminated from the ceramic wall due to the difference in densification behavior of metal and ceramic tape In the sintering process of alumina-glass ceramic, the melting of glass enhances the sintering of alumina at lower temperatures due to liquid phase sintering. The flux of glass from the alumina to refractory metals increases the addhison of the metal/ceramic joint. In contrast to refractory metals, in the Pt/glass system, due to the complex surface tension behavior, increasing the temperature increases the flow of glass at first but the decrease in the Pt/glass surface tension decreases the flow and causes the seperation of the glass from the Pt as shown in figure 2.

Point 3 in figure 3 shows the average composition of the via. It is mostly occupied with platinum and has aluminum, carbon and oxygen as impurity. Presence of carbon could be attributed to an incomplete burn-out

process. Aluminum and oxygen could be from the diffusion of alumina in the platinum in high temperature. However, the presence of all three elements could also be due environmental contamination and experimental error.

The elemental analyses of point 1 and 2 indicated the presence of Si, Mg and Ca in addition to Pt, Al, C and O. This elemental composition implies the formation of the glass on the surface of platinum via. Glass particles usually have the flake-like structure with the average size of 10-20 µm, but the glass particles shown in figure 3 have spherical shape with size of 5 µm. This indicates the melting of glass at high temperatures. At high temperatures, melted glass failed to wet the platinum and floated to the surface of the via, and solidified into the spherical shape in the cooling process. In the cool down process, the temperature of the surface of platinum is less than the bulk. This temperature gradient could differentiate the glass/metal surface tension and produces capillary force to push the molten glass out of the metal. This mechanism can explain the formation of glass on the top surface of platinum via.



Figure 3: Backscatter image of Platinum Feedthrough fired in Air

### 3.3. Via diameter

Similar to glass particles, metal oxide materials, such as alumina powder, which are used to decrease the shrinkage of the ink, could also migrate to the surface of the via due to the capillary force. This migration increases by increasing the aspect ratio of the via. Via diameter could control the migration of the light oxide material to the surface since increasing the via diameter decreases aspect ratio. Results of the application of 20% volume fraction of Al<sub>2</sub>O<sub>2</sub> in the platinum ink, for the vias with different diameters fired in air atmosphere, are shown in figure 4.

As depicted in the figure 4, increasing the via diameter decreases the migration of the oxide to the surface. The surface of the 100 µm via diameter completely covered with recrystized ceramic. The EDS results show that the ceramic has the composition of the alumina.

100 µm



Figure 4: Microstructures of Platinum via with, 20% Al<sub>2</sub>O<sub>2</sub> in different via size

### 3.4. Effect of Atmosphere

Atmosphere condition is the most important factor to control the interface of platinum and alumina. In addition to that replacing air with neutral atmosphere, such as argon, could eliminate evaporation of platinum. SEM image of sample fired in the air and Ar atmosphere with heating rate of 5 °C/min are showed in figure 5.

As it can be seen that platinum densified in Ar more than air. This behavior of platinum could be attributed to the decomposition of platinum dioxide and desorption of oxygen from the bulk of platinum. This oxygen layer is adsorbed on the layer of platinum below the surface and it is almost inert in temperature below 800



**Figure 5:** Secondary electron SEM image of feedthrough fired in air and argon atmosphere.

°C [21]. The results of DSC experiments of sintering of platinum are shown in figure 6. The colorimetric experiment followed the same condition was used in firing of feedthrough. As it can be seen, the exothermic reaction of decomposition occurred around 1000 – 1200°C. Heat generation in neutral atmosphere is more than air, which could result in local super heating in sample and increase the shrinkage of the platinum.



**Figure 6:** DSC analyses of the platinum powder in air and argon.

#### 3.5. Pt/Alumina reaction

The reaction of  $Pt/Al_2O_3$  could be enhanced in a reduced atmosphere due to Pt-Al alloy formation [15, 22]. Hydrogen can reduce alumina and the major product of this reaction is  $Pt_3Al$ . The X-ray diffraction pattern



**Figure 7:** X-ray diffraction pattern of Platinum/ Alumina combination heated in two different environments in 1400°C

of reaction of the 25% Vol. powder mixture of alumina/platinum in the hydrogen and air atmosphere in 1400°C is shown in figure 7.

As is seen form figure 7, the first peak of  $Pt_3Al$  appered in  $2\theta=10.40^\circ$  in a hydrogen atmosphere, which indicates the reation between platinum and alumina. To better understand the reaction of Pt/Alumina, a high temperature X-ray diffraction technique was utilized, using carbon as reducing agent. The result of the reaction of Pt/Alumina in the prescence of carbon is shown in figure 8.



Figure 8: High temperature diffraction data of Pt/Alumian system

The first two peaks in  $2\theta$ =7.77° and 8.35° appeared at the temperature about 530 °C. Those peaks are related to tetragonal Ir, Si structure. Pt, Al in temperatures lower than 400 °C exhibit a tetragonal crystal structure [23]. By increasing the temperature up to 650 °C, another peak appeared at  $2\theta$ =6.65°. That peak is attributed to the formation of rombohedral structure of aluminum carbide [24]. In the temperature above 1250 °C, a new peak appeared at  $2\theta=7.15^\circ$ , which corresponds to the cubic Pt, Al structure. The Ir, Si tetragonal structure could transform to cubic Cu<sub>2</sub>Al easily. The Pt<sub>2</sub>Al was formed with the tetragonal structure at low temperature and the crystal structure transformed to the cubic structure at high temperatures. From the results showed in figure 7, the equilibrium structure of the Pt<sub>3</sub>Al is cubic, with sufficient amount of time and hydrogen concentration, the tetragonal structure transforms completely to the cubic structure.

A common firing profile consists of heating and soaking in temperature below 450 °C to burn-out the organic. During this portion of the profile, polymeric binder produces a fair amount carbon, which could evaporate from the sample. Different burn-out process could change the carbon evaporation behavior and subsequently change the possibility of the reaction in platinum/carbon/alumina system. The results of firing of platinum feedthrough with two different burn-out process is shown in figure 9.

It could be seen that the thickness of ceramic layer around platinum decreases with increasing the time of burn-out process. Fast burn-out process could decrease the evaporation of the carbon from the system and increase the reaction between platinum and alumina.

TEM image of the platinum/alumina interface showed in figure 10. The intfrace shows strong bonding between platinum and alumina, which is due to the rduction reaction of alumina nd formation of PtAl<sub>3</sub>.



**Figure 9:** SEM image of the platinum particle via fired in different burn-out prcosee

#### 3.6. Firing temperature

Slow

Fast

In addition to the reaction products, temperature could control the final microstructure of the feedthrough sample as well. The effect of firing temperature on the properties of the Pt-100-10 nano platinum powder feedthrough in a reduced atmosphere is given in figures 11. After the burn-out process, each feedthrough was heated up to the desired temperature at the heating rate of 10 °C/min, maintained at that temperature for an hour and was rapidly cooled down. Firing was done in the H<sub>2</sub> – Ar (25-75) atmosphere.



**Figure 10:** TEM image of the interface between platinum via nd ceramic particle.



**Figure 11:** SEM image of the platinum particle via fired in different temperature

The behavior of platinum powders starts to change after the temperature reaches 1350 °C. In the range of 1350 – 1550 °C, by increasing the temperature, platinum starts to expand and fill the via. In the temperature range of 1350 – 1450 °C, a coating of alumina covers the surface. In this temperature range, platinum dissolves some of the ceramic from the sidewalls and the alumina is transported to the top surface. At 1450 °C, the alumina covers the surface completely with a large grain structure. At the highest temperature, platinum completely dissolves the ceramic and there is no sign of it on the surface. A large droplet shape ceramic also appears on top of the platinum via, but it does not have a grain structure. The ceramic structure is more like a solidified glass, which was confirmed from the EDS results.

The results of the microsized platinum powder feedthrough fired in 1550 °C in the same condition is showed in figure 12 for comparison.



**Figure 12:** SEM image of the micron-sized platinum particle feedthrough via fired in 1550°C

By increasing the platinum particle size, platinum starts to expand and ultimately makes a dome-like structure above the via. As is seen from figure 12, the ceramic around the platinum disappears and creates a recessed structure. At 1550 °C, the platinum via is 150  $\mu$ m in diameter and the recessed area around the platinum increased up to 200  $\mu$ m. Platinum dissolves alumina and glass from the surrounding area and expands. By the introduction of alumina and glass particles into platinum, platinum starts to expand up to 150  $\mu$ m and creates a dome structure. Platinum powder dissolves alumina at high temperatures, however, different final microstructure of the two platinum particles could suggest that smaller size particles wicks the alumina and push it to the top surface by capillary action.

Figure 13 shown the concentration gradient of different elements in platinum for the micrion-sized platinum powder via fired at 1550 °C, detremiuned by EDS analyses. It is obvious that silicon diffuse through the entire width of the via. The weight concentration of the silicon is almost constant and is about 3.5%. Pt-Si phase diagram [25] does not show any solid solution of silicon of silicon in platinum. Therefore, the 3.5% concentration of silicon in platinum suggests the reaction of SiO<sub>2</sub> with platinum and the creation of a Pt<sub>3</sub>Si phase. Silicate compounds, which are used mostly as the glass phase in HTCC ceramic tape formulation, could be the source of SiO<sub>2</sub> for reaction. Aluminum is foundin the first few microns from the interface. The concentration of aluminum in platinum is about 1.5 Wt.%, which is equal to the solubility of aluminum into platinum [26]. After about 8 microns from the interface, the concentration of the alumina dropped drastically and it is almost negligible. This behavior could suggest that silicon prevents diffusion of aluminum into platinum.



**Figure 13:** Concentrations gradient of different elements in platinum for the micron-sized platinum powder. Feedthrough fired in the  $H_2$  – Ar (25-75) atmosphere at 1550 °C

To minimize the effect of silicon on the diffusion of the aluminum, the 99.9% HTCC ceramic tape were use to develop a feedthrough and the concentration gradient of different elements are shown in figure 14.



**Figure 14:** Concentration gradient of different element in platinum for the micron-sized platinum powder and 99.9% HTCC ceramic tape. Feedthrough fired in the  $H_2$ – Ar (25-75) atmosphere at 1550 °C

It is clear that the concentration of the aluminum gradually decrease, however the final concentration is about 5 Wt%, which is more than the solubility limit of the aluminum in platinum. This behavior promotes the formation of Pt<sub>3</sub>Al phase. The concentration of oxygen shown in Figure 7 and 8 is almost zero in all cases, which is due to the reduction of alumina and silica in the reduced atmosphere. It should be noted that the EDS does not show any trace of platinum in alumina, which indicates that diffusion only occursfrom the alumina side to the platinum.

At high temperatures, platinum absorbs the ceramic grains and glass from the sidewalls and that expands the structure. Platinum dissolves silicon and cannot maintain the solubility of the aluminum, thus the aluminum moves to the surface as alumina. Based on the temperature, alumina could recrystallize to large grains or small grain size. However, at temperatures above 1500 °C, it dissolves into the platinum and remains as a reaction product. This could be due to the formation of a Pt-Al compound as a second phase in the platinum-alumina interface.

### 4. Conclusion

The results of firing of feedthrough assembly in different conditions showed that glass migration mechanism is complicated in Pt/Al<sub>2</sub>O<sub>3</sub> system and glass is not very useful in improving the metal/ceramic joint. Introducing some metal oxide powder as a shrinkage control agent to the formulation of the platinum ink also could improve the sintering kinetics of platinum and improve the properties of feedthrough assembly. However, the oxide material could float to the surface of the via. Decreasing the via diameter increases the migration of the ceramic powder to the top surface of the via. Mixture of hydrogen and argon is the best possible environment for firing the feedthrough. In addition to that, pt/alumina mixture starts to react in temperature as low as 550 °C. The tetragonal Pt<sub>3</sub>Al forms in low temperature and transforms to the stable cubic structure in high temperature. In reduced atmospheres, the firing temperature could be adjusted by the size of platinum particle. Larger platinum particles could be fired in temperatures lower than 1550 °C.

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### Mechanical Properties of Low Temperature Co-fired Ceramics: Testing Methodologies for Strength Characterization

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**Abstract:** Functional components such as multilayer Low Temperature Co-fired Ceramics (LTCC) are examples of combination of a ceramic-based substrate with internal electrodes as well as surface features (e.g. metallization, contacting pads, cylindrical vias, etc) employed to provide the component with a given functionality. Due to miniaturization and design complexity, no standard methods for mechanical testing can be applied for component characterization. An experimental approach is here presented using the Ball-on-three-Balls (B3B) method, based on localized biaxial testing of metalized LTCC samples, at several locations under different environments, to quantify the effect of surface features or metallization (e.g. contacting pads or cylindrical vias) on the material strength distribution. Experimental findings show that the strength distribution of LTCC can be negatively affected by environmental degradation, where Subcritical Crack Growth (SCCG) phenomena can be enhanced in conditions of high relative humidity. In addition, metallization at the surface subjected to tensile stresses can even raise the strength of the component acting as a protective layer against environmental degradation, whereas cylindrical vias can become weak points in the design.

Key words: Functional ceramic components, Biaxial Strenght, Fracture, Weibull Statistics, Environmental brittleness.

### Mehanske lastnosti keramike z nizko temperaturo žganja: metodologije testiranja trdnosti

**Povzetek:** Funkcionalne sestavine, kot je večplastna keramika z nizko temperaturo žganja (LTCC), so primeri kombinacij substratov na osnovi keramike in internih elektrod, kot tudi površinskih lastnosti (metalizacija, kontaktne blazinice, cilindrične povezave itd.), ki dajejo komponenti željeno funkcionalnost. Standardne metode testiranja mehanskih lastnosti, zaradi miniaturizacije in kompleksnosti oblik, niso primerne za karakterizacijo elementa. V članku je predstavljen je eksperimentalen postopek na osnovi Ball-on-three-Balls (B3B) metode, ki temelji na lokaliziranem dvoosnem testiranju metaliziranih LTCC vzorcev na različnih lokacijah in okoljih in je namenjen kvalifikaciji vpliva površinskih lastnosti ali metalizacije (kontaktne blazinice, cilindrične povezave) na porazdelitev sil v materialu. Rezultati poskusov so pokazali, da ima lahko degradacija zaradi okolja negativen vpliv na razporeditev sil LTCC. Pri visoki relativni vlažnosti se lahko pospeši rast razpok (Subcritical Crack Growth – SCCG). Dodatno lahko površinske natezne sile zaradi metalizacije izboljšajo čvrstost in delujejo kot zaščitni sloj pred vplivi okolja, pri čemer pa cilindrične povezave predstavljajo šibke točke v konstrukciji.

Ključne besede: funkcionalne keramične komponente, dvoosna sila, frakcije, Weibullova statistika, okoljska občutljivost.

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### 1. Introduction

Many applications in microelectronics involve combinations of ceramic and metal constituents. Due to the different properties of the materials involved (e.g. thermal expansion coefficients, elastic constants, yield strength) components can be subject to internal stresses during fabrication, which may induce cracks that truncate the electrical performance of the device [1-4]. Other processes after fabrication (e.g. packaging) can also involve high tensile stresses which may damage the component. In addition, mechanical stresses occurring during service conditions (e.g. rapid temperature changes, mechanical vibration, bending) can affect the structural integrity of the device.

Functional components such as multilayer varistors (MLV), piezoelectric actuators (MPA), Low Temperature Co-fired Ceramics (LTCC) or semiconductors, among others, are examples of combination of a ceramicbased (or Si-based) substrate with internal electrodes as well as surface features (e.g. metallization, contacting pads, cylindrical vias, etc) employed to provide the component with a given functionality. For instance, LTCC consist of a complex three-dimensional micronetwork of metal structures embedded within a ceramic substrate with a large content of glass. The low sintering temperature of the ceramics in LTCC (i.e. below 900 °C) can be achieved by using a glass matrix with a low melting point, allowing a vitrification of the glass ceramic composite material [5, 6]. This makes feasible the use of excellent electronic conductors such as silver, gold or mixtures of silver-palladium, arranged within and/or on the surfaces of the ceramic substrate, forming complex multi-layered structures. Today, they can be found in electronic devices (e.g. for mobile and automotive technologies) which have to operate under harsh conditions such as relatively high temperatures and mechanical shock under different environments.

A limiting factor for the lifetime of glass- and ceramicbased components is associated with the subcritical crack growth (SCCG) phenomenon, especially in environments with high moisture content [7-9]. The lifetime is thus related to their mechanical strength and crack growth resistance during service conditions. It is also known that metallization as well as surface features (such as contact pads or metal cavities) can affect the strength of components (see for instance [10-13]). In summary, a combined effect of environmental degradation and geometry features can decrease the strength of brittle functional components.

In this paper, a testing method (the Ball-on-threeballs) is employed to measure the mechanical biaxial strength of commercial and sample LTCCs. Different loading conditions and environments are selected aiming to reproduce possible loading scenarios during service. In addition, an experimental approach combining both humid and relatively dry environments (i.e. water and silicone oil) is presented to assess the effect of different surface metallization on the strength of sample LTCC components.

### 2. Experimental

2.1. Material of study

The substrate (glass-ceramic) employed to fabricate the LTCC specimens of study is made of approx. 50%

of  $Al_2O_3$  as filler and 50% of several glasses containing Ca, Na, Si, K, B and Al, where the crystallisation degree after sintering exceeds 90%. Figure 1 shows a SEM micrograph of a typical polished area on a LTCC of study. The bright phase corresponds to  $Al_2O_3$  particles which are embedded in a glassy matrix (grey phase). Microstructural characterisation of the bulk material can be found elsewhere [14].



**Figure 1:** SEM micrograph of a LTCC microstructure showing alumina phase (bright) in a glassy matrix (dark).

### 2.1.1. Commercial LTCC components

The specimens used for the biaxial strength tests were cut from commercial LTCC-Tapes (panels of ca. 100 x 100 x 0.45 mm<sup>3</sup>), provided by the company TDK-EPC, Deutschlandsberg, Austria (Fig. 2).



**Figure 2:** Scheme of a panel where the samples are cut out in such a way that the position to be tested lies in the centre of the specimen.

For the inner metallisation only Ag is used, whereas for the outer metallisation the silver pads are covered with a nanometric Ni/Au layer, which is employed to avoid corrosion and further guarantee good adhesion of possible soldered components. The thickness of the metal pads is approx. 7  $\mu$ m. The processing route employed was tape casting and the co-sintering was performed in a furnace at 850 °C. After sintering, four different samples of rectangular plates of ca. 11.0 x 9.7 x 0.45 mm<sup>3</sup> were cut from each panel, as indicated in Fig. 2. In each case, the centre of the plate contains a different region of the LTCC-unit. Bulk specimens of the same dimension were also tested as reference material.

### 2.1.2. Sample LTCC

In order to distinguish the effect of inner structures and surface metallization on the biaxial strength, sample structures were fabricated using the same process as for commercial LTCC components. Rectangular testing plates of *ca.* 11.0 x 9.7 x 0.45 mm<sup>3</sup> were diced from each panel with a diamond saw. In addition to bulk material without metallization as reference material (Fig. 3a), three LTCC samples with different surface metallization were fabricated:

- MT: Bulk LTCC with a full metallization layer of ca.
   10µm on top of the substrate (Fig. 3b).
- (ii) CP: Bulk LTCC with contact pads of 1.5 x 1.8 mm and ca. 10µm thickness, covering the 75% of the top surface (Fig. 3c).
- (iii) CV: Bulk LTCC with cylindrical vias on the top with a diameter of 100μm and a pitch of 300μm, with a depth into the material of ca. 50μm (Fig. 3d).

#### 2.2. Mechanical testing: Ball-on-three-balls

The biaxial strength of the LTCC specimens was determined using a miniaturised Ball-on-three-Balls (B3B) fixture (Fig. 4a) especially built in-house to match the dimensions of the supplied components. In the B3B method, a rectangular plate (or a disc) is symmetrically supported by three balls on one face and loaded by a fourth ball in the centre of the opposite face (see Fig. 4b), which produces a very well defined biaxial stress field [15, 16]. The four balls had a diameter of 8 mm. A pre-load of 7 N was applied to hold the specimen between the four balls. The load is increased until fracture occurs, and the fracture load can be used to calculate the maximum tensile biaxial stress in the specimen at the moment of fracture.

For a bulk plate of an elastically isotropic material the equivalent maximum stress  $\sigma_{max}$  corresponding to the fracture load *P* can be calculated as follows:

$$\sigma_{\rm max} = f \cdot P/t^2 \tag{1}$$



**Figure 3:** Samples with different metallization at the top surface of the substrate: a) bulk without metallization, b) bulk with full metallization layer, c) bulk with contact pads, and d) bulk with cylindrical vias of 100µm diameter and 50µm depth.



**Figure 4:** a) Schematic of the ball-on-three-balls test for biaxial testing and b) FE simulation of the stress distribution in the plate during loading.

where *t* is the specimen thickness, and *f* is a dimensionless factor which depends on the geometry of the specimen, on the Poisson's ratio of the tested material, and on the details of the load transfer from the jig into the specimen. Elastic properties were determined by means of the Resonant Beam Method [17] in a bulk LTCC sample, resulting in a Young's modulus of  $E = 113 \pm 1$  GPa and a Poisson's ratio of  $v = 0.2 \pm 0.01$ . The factor *f* was calculated for the given geometry and Poisson's ratio as function of the thickness of the specimens. The resulting geometric factors corresponding to commercial LTCC and sample structures investigated here are reported elsewhere [13, 18].

#### 2.2.1. Testing of commercial LTCC

The tests on commercial LTCC were conducted under displacement control at a rate of 0.5 mm/min and a relative humidity (RH) of  $23 \pm 2$  % at  $21 \pm 1$  °C (i.e.  $H_{abs} \approx 4.3 \pm 0.4 \text{ g}_{H20}/\text{m}^3$ ), using a universal testing machine (Zwick Z010, Zwick/Roell, Ulm, Germany) with a load cell of 200 N. The load was increased until fracture occurred and the fracture load was used to calculate the maximum tensile biaxial stress in the specimen at the moment of fracture using Eq. (1). A total of 30 specimens were tested for each series.

### 2.2.2. Testing of sample structures

In order to discern the effect of surface metallization (e.g. contacting pads or cylindrical vias) and the effect of the environment on the strength of components, a novel experimental approach has been proposed. Biaxial strength tests were performed within two different environmental conditions: a) in water and b) in silicone oil bath, in order to reproduce high and low relative humidity conditions, respectively. In both cases, the samples (as described in Fig. 3), were placed in the B3B fixture and the corresponding fluid (i.e. water or silicone oil) was pumped into the specimen holder to fully cover the specimen during the entire loading. The temperature was maintained constant for all tests, i.e. 22  $\pm$ 2 °C. The number of specimens tested for each sample ranged between 15 and 30, for statistical significance. The tests were conducted under displacement control at a constant cross-head speed (i.e. 0.1 mm/min) using a universal testing machine, (Zwick Z010, Zwick/Roell, Ulm, Germany).

### 3. Results and discussion

#### 3.1. Strength of commercial components

Fig. 5 shows a Weibull diagram of the four LTCC series corresponding to four different tested regions of the LTCC-unit. Bulk specimens of the same dimension were also tested as reference material. Each distribution was collected on a sample of 30 specimens, which ensures statistical significance for the Weibull analysis. The nominal failure load, *P*, is represented vs. the probability of failure, *F*. The corresponding equivalent failure stress,  $\sigma_{r}$  calculated with Eq. (1) for every tested specimen is also represented in the plot. The strength results from the bulk specimens are also shown for comparison.

It can be inferred from Fig. 5 that all series follow a Weibull distribution. Nevertheless, the strength distribution between some of the series with metallisation and the bulk series is different. This can be associated with stress concentrations near metallization regions and/or residual stresses developed during co-sintering of metal-ceramic parts. In addition, some of the diagrams seem to follow a typical two-parameter Weibull distribution (*e.g.* series 1 in Fig. 5), whereas others show a so-called "concave banana-shape" (*i.e.* the strength distribution in the Weibull diagram deviates from a lineal trend, pointing downwards for lower failure stress values). In such cases (see for instance series 2 and 4 in Fig. 5) a minimum failure stress level ("threshold stress") may be defined under which the material may not fail. This can be a result of metal electrodes arresting the crack in its propagation from the surface towards the bulk. A comprehensive study of the crack propagation can be found in [11, 13].



**Figure 5:** Weibull diagram of LTCC samples corresponding to four different tested regions of the LTCC-unit. A sample of bulk specimens is also shown for comparison.

### 3.2. Effect of surface metallization on strength distribution

In order to discern the effect of surface metallization (e.g. contacting pads or cylindrical vias) and the effect of the environment, an experimental approach has been proposed, based on localized biaxial testing of brittle samples at several locations under low and high humidity environments (i.e. silicone oil and water) respectively [18]. Figure 6 represents the corresponding Weibull diagram, where the equivalent failure stress,

 $\sigma_r$  (calculated for every specimen according to Eq. (1)) is represented versus the probability of failure, F, for the bulk sample tested in water and in silicone oil. It can be observed that the failure stress values follow a Weibull distribution, which is associated with the flaw size distribution in the sample. The Weibull parameters,  $\sigma$  and m were calculated according to EN-843-5 [19]. The full line represents the best fit of the strength data using the maximum likelihood method. The 90% confidence interval for the measured values is also shown in the referred figure as broken lines. It represents the range where the true Weibull parameter can be found with a 90% probability and reflects the influence of the sampling procedure. The characteristic flexural strength and Weibull modulus for all samples tested, with the calculated 90% confidence intervals, are given in Table 1.



**Figure 6:** Weibull diagram of bulk LTCC samples tested in water and in oil.

**Table 1:** Characteristic strength,  $\sigma_0$ , and Weibull modulus, *m*, of samples with different surface metallization, tested in water and in silicone oil.

Sample	Water		Silicor	ne Oil
	$\sigma_{_0}^{}$ (MPa)	m	$\sigma_{_0}$ (MPa)	m
Bulk	257	32	309	41
	(253–261)	(20–42)	(305–313)	(26–54)
MT	303	20	305	27
	(295–311)	(13–26)	(300–311)	(17–35)
СР	296	22	298	38
	(289–303)	(14–29)	(294–302)	(24–50)
CV	164	28	220	42
	(161–167)	(17–37)	(217–222)	(27–54)

The effect of the environment on  $\sigma_0$  can be clearly seen in Fig. 6, as has been reported elsewhere [14]. The SCCG of defects during loading in water diminishes the strength (ca. 20%) as compared to testing in dry conditions for the same loading rate. According to the results listed in Table 1, for the case of samples with a metal layer on the top (tensile) surface (MT samples), no difference can be appreciated between both environments, yielding strength values similar to those for bulk substrate tested in oil (i.e. 309 MPa). This indicates that the metal layer "protects" the substrate from SCCG effects. The same applies for CP samples loaded both in water and in oil (see Ref. 18 for more details). A different situation is found for CV samples which reveal not only an effect of the environment (i.e.  $\sigma_0 = 165$  MPa in water vs.  $\sigma_0 = 220$  MPa in oil), but also a negative effect of the vias on the strength, compared to substrate (bulk) material (see Table 1); i.e. strength can be reduced up to 40% compared to bulk material.

### 4. Conclusions

In summary, it has been shown that localised biaxial testing using the ball-on-three-balls method performed in different environments can be used as methodology for reliability assessment as well as to quantify the effect of surface features on the strength of functional ceramic components. Whereas surface contacts such as cylindrical vias can become weak points in the design, metallization at the surface subjected to tensile stresses can even raise the strength of the component acting as a protective layer against environmental degradation.

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### LTCC-Based Sensors for Mechanical Quantities

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**Abstract:** Besides their excellent dielectric and thermo-mechanical characteristics Low Temperature Cofiring Ceramics (LTCC) are also well suited for the fabrication of 3D micromechanical components such as sensors for mechanical quantities. This paper describes the development of such sensors covering some material and technological aspects. Furthermore, the design process for mechanical sensors is discussed as well as application examples of sensors for the detection of pressure, force, acceleration and flow.

Key words: LTCC, Sensors, 3D-Integration.

### Senzorji mehanskih veličin na osnovi LTCC

**Povzetek:** Poleg odličnih dielektričnih in termo-mehanskih lastnosti so keramike z nizko temperaturo žganja (LTCC) primerne tudi za izdelavo 3D mikro mehanskih komponent, kot so senzorji mehanskih veličin. Članek opisuje razvoj teh senzorjev vključno z nekaterimi tehnološkimi in materialnimi vidiki. Dodatno je predstavljen postopek oblikovanja mehaničnih senzorjev in nekaj primerov senzorjev tlaka, sile, pospeška in pretoka.

Ključne besede: LTCC, senzorji, 3D-integracija

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### 1. Introduction

Ceramic sensors are applied when specific requirements have to be fulfilled, e.g. a high reliability at elevated and cycled temperatures, harsh environment as well as in aggressive chemicals.

The ceramic multilayer technology (e.g. LTCC) enhances these advantages because of its ability (i) for a complex 3D miniaturization with embedded deformable bodies (cantilever, diaphragms), channels and cavities as well as the ability (ii) for the direct integration of electronic components for signal conditioning and processing.

One reason for the outstanding commercialization success of LTCC-based sensors is the cost level which is mainly defined by material and process costs. In order to reduce material and process costs, miniaturization is the most important leverage.

### 2. Material aspects

The manufacturing of LTCC-based 3D micro-components means the co- and post-firing of glass-ceramic

layers and different functional materials. During cofiring, in particular, intensive mechanical and chemical interactions can appear, strongly influencing the component performance.

The successful processing of multilayered multi-material based miniaturized LTCC components requires the proper control of different materials and technological aspects.

### 2.1 LTCC

One reason that ceramic materials are advantageously used for deforming bodies in sensor applications is their linear stress vs. strain behaviour. Table 1 compares different ceramic materials in terms of their mechanical properties. **Table 1:** Mechanical properties of different ceramics(CTE ... coefficient of thermal expansion (20... 400°C), E... Young's modulus,  $\sigma_{\rm B}$  ... bending strength).

Material	CTE	E	$\sigma_{_{B}}$	$\sigma_{_{\rm B}}/{ m E}$
	ppm/ K	GPa	MPa	10 <sup>-3</sup>
96% Al <sub>2</sub> O <sub>3</sub> <sup>1)</sup>	7.6	350	310	0.9
99.8% Al <sub>2</sub> O <sub>3</sub> <sup>2)</sup>	7.5	406	630	1.6
LTCC 3)	5.8	120	320	2.7
YSZ <sup>4)</sup>	11.2	210	1050	5.0
ZTA <sup>5)</sup>	8.1	357	1350	3.8

Datasheet values: <sup>1)</sup> CeramTec V38, <sup>2)</sup> CeramTec RK 87, <sup>3)</sup> Du Pont DP 951, <sup>4)</sup> CeramTec MZ 429, <sup>5)</sup> CeramTec DC 25.

The  $\sigma_{\rm g}$ /Eratio determines the dimension of the deforming bodies in terms of sensitivity and overload stability. The larger the  $\sigma_{\rm g}$ /E ratio the smaller the deforming bodies can be designed. It can be seen that LTCC is well suited because of a  $\sigma_{\rm g}$ /E ratio of 2.7. However, YSZ ( $\sigma_{\rm g}$ /E = 5) and ZTA ( $\sigma_{\rm g}$ /E = 3.8) have a better mechanical performance, but embedding e.g. of low sintering noble metals as well as resistors can only be realized using a low temperature firing system.

Different types of LTCC show a different bending strength behavior [1]. Best values can be obtained using the Du Pont's Green Tape 951 system (Figure 1). It must however be noted that, in order to ensure longterm reliability, static [2] and cyclic fatigue [3] must also be accounted for.



**Figure 1:** Comparison of different LTCC types regarding their fractural strength (re-calculated after [1]).

The 951 system offers different tape thicknesses (50 – 254  $\mu$ m unfired) as well as a full system of the required pastes (inner/outer conductors, via, outer/inner resistors). This makes it particularly well suited for the fabrication of sensors.

The shrinkage control of LTCC multilayer components plays an important role regarding the final dimensional control e.g. for advanced electronic packages (chip sized packages, flip chip) but also in the case of mechanical sensors.

Shrinkage occurring during firing can be influenced by controlling the process factors (of lamination and firing). Figure 2 shows the influence of lamination temperature and pressure.



**Figure 2:** LTCC DP 951 X-Y shrinkage  $(S_{X-Y})$  vs. lamination temperature (T) and pressure (p) [4].

### 2.2 Functional Thick Films

#### 2.2.1 Metallization

In many cases, LTCC-suited inner metallization pastes are silver-based. The interaction between silver and LTCC during co-firing was described in the past by several authors e.g. [4-6], it can lead to significant warping of thin and 3D structured LTCC geometries.



**Figure 4:** LTCC DP 951 X-Y shrinkage  $(S_{x-Y})$  vs. heat up ramp (HR) and metallization degree (MD) [4].

Figure 4 shows that the shrinkage of a DP 951-based LTCC multilayer during firing is – in addition to the lamination parameters – also influenced by the degree of metallization and the heat up ramp. It can be seen that there is only a slight effect on the X-Y shrinkage by changing the heat up rate, when the multilayer is not metalized. In the case of an extensive metallization (60% metallization area) there is a strong dependence on the heat up ramp. The metalized multilayer shrinks in the same way as the non-metalized multilayer at higher heat up ramps (12.8% @ 8K/min). Using low heat up ramps (2 K/min), the shrinkage is reduced to 12.1%. The silver layer has a "locking" function due to the different shrinkage behavior.

Besides this pure mechanical effect there are also chemical interactions between the silver layer and the LTCC. [6] and [5] demonstrated a strong degree of silver diffusion into the LTCC, which can reach some 10  $\mu$ m. [7] showed that silver influences the viscosity of the LTCC glass leading to an earlier densification and crystallization. Because silver enters the LTCC glass matrix in an oxidized status, a nitrogen sintering atmosphere can minimize the discussed effects, by shifting the chemical equilibrium between Ag<sup>+</sup>(glass) and Ag0 (metal) in favour of the latter.

#### 2.2.2 Piezo-Resistors

The transformation of a mechanical quantity into an electrical signal by piezo-resistors is one of the most common measuring principles. Using a deformable body with piezo-resistors in the areas with high mechanical strain, a mechanical input quantity (e.g. force) can be transduced into a proportional resistance/ bridge voltage change (Figure 5).



Figure 5: Sensor transmission chain [8].

Based on their microstructure and effective conducting mechanism, thick-film resistors (TFRs) always have a strain sensitive behavior.

As well as the thermal behavior, the strain sensitivity of TFRs is dominated by insulating/semi-conductive nanoscale glass layers between conducting particles, within the three-dimensional conductive chains, straindependent interparticle tunneling being the favoured mechanism [9, 10]. The strain sensitivity is specified using the K- or gauge factor which determines the ratio between the relative change of resistance and the applied strain.

Measured gauge factors of TFRs are between 2 and 35. They are influenced by (i) the composition of the TFR (type, grain sizes and proportion of glasses and conductive phases), (ii) the firing process (dissolution of the conductive phase into the glass), and (iii) the interactions between the TFR and the substrate as well as the terminations (diffusion of silver ions into the TFR glass matrix) [11]. The maximum signal gain of TFRs is limited by noise. Therefore, the effective signal-tonoise ratio is suitable as a normalized parameter [12]:

$$SNR_{eff}[dB] = -20 \cdot \frac{\lg U_s}{U_N}$$
 (1)

 $U_{\rm s}$  and  $U_{\rm N}$  are the effective signal voltage and the peakto-peak noise voltage. Here, *U*S was calculated using the measured gauge factors, a supply voltage of 5 V and a relative strain of  $e = 2.7 \cdot 10^{-4}$  (corresponds to 50% of the maximum deflection before substrate breakage).  $U_{\rm N}$  was calculated by solving the definition of the noise index (NI).



**Figure 6:** Strain sensor paste development [12].Noise index (NI) vs. BET surface area of RuO<sub>2</sub>.

In order to improve the  $SNR_{\rm eff}$  [12], compositions were developed as a mixture from  ${\rm RuO}_2$  (four different particle size distributions, one mixture) as well as two different glasses (five different particle sizes). The aim of these investigations was a TFR sheet resistance of 10 kOhm/sqr.

The results showed that the noise index (NI) and the gauge factor of TFRs are strongly influenced by the particle size distribution of the glasses as well as the conductive phase. Figures 6 and 7 illustrate that there is a

compromise between gauge factor and noise behavior of TFRs.



**Figure 7:** Strain sensor paste development [12]. Longitudinal gauge factor vs. BET surface area of RuO<sub>2</sub>.

However, these developments cannot improve the DP 2041 characteristics regarding the *SNI*<sub>eff</sub>.

Further activities are planned concerning the development of new glass types, the optimization of firing and the analysis of alternative conductive phases (pyrochlores).



**Figure 8:** Effective signal-to-noise ratio (SNReff) [12]. Comparison of IKTS and commercial pastes.

### 2.2.3 Sacrificial Materials

Sacrificial materials temporarily stabilize functional elements during lamination and sintering. They are removed during or after sintering. Two types of sacrificial materials are differentiated:

- Mineral sacrificial paste (MSP),
- Fugitive sacrificial paste (FSP).

MSP consists of glass, an active substance and vehicle [13, 14]. The active substance is a mineral filling material. After sintering, the MSP provides a solid layer and can be removed with aqueous acetic acid. MSP suits well for cantilevers and freestanding structures with high level requirements regarding the accuracy. Closed structures are difficult to handle as MSP has to be dissolved with acid.

FSP consists of organic and graphite powder and contains no glass phases [15, 16]. The graphite is oxidized to carbon dioxide at about 700 °C. Thus, it is well suited for closed cavities, channels and diaphragms in LTCC because FSP volatilizes through the porous LTCC or open channels during sintering [15].

In order to stabilize embedded mechanical structures during sintering, FSP oxidation must be prevented using a non-oxidizing sintering atmosphere (e.g. nitrogen). In this case, a "vent" is required for out gassing the carbon dioxide from the embedded structures. The appropriate timing for the change from oxygen to nitrogen and vice versa is a critical issue. It was studied for DP 951 in [17].

The resulting profile is shown in Figure 9. The amount of FSP and the geometry of the embedded structures require an adjustment of the sintering procedure for every component to avoid warping and FSP residue effects.



**Figure 9:** Customized sintering profile for surviving FSP after sintering DP 951 [17].

However, sacrificial materials often require the application of zero-shrinkage techniques [18] because of their unadapted shrinkage behavior in comparison to standard LTCC materials.

### 3. Technological aspects

The higher functional integration in multilayer-based sensors and microsystems complicates their processing compared to that of multilayer substrates for electronic assemblies. Sensors for mechanical quantities, in particular, need (i) functional structures like freestanding deforming bodies, fluidic channels or heating bridges which have to be processed with no sagging or warping, (ii) deforming bodies with reinforced regions for seismic masses or force application, and (iii) low dimensional tolerances of the processed substrates to provide low variances of the performance of the fabricated sensor elements. It is a challenge to customize the standard LTCC technology for these aspects. However, the technology varies according to design, material and dimension of the sensor elements.

### 3.1 Sagging and warping

Sagging and warping are mainly caused by the lamination and sintering process. Below, some techniques are mentioned in order to reduce these imperfections:

- Cold lamination (uniaxial) at 30 °C reduces sagging.
- An isostatic pre-lamination of the unstructured tapes provides higher stability for the following processes like lamination. However, higher pressure ranges will then be necessary in the following lamination steps.
- A differential shrinkage of several single layers can be achieved by pre-laminating which helps to minimize sagging effects at thin deforming elements (diaphragms, cantilevers) during firing (Figure 10).
- Fixation and support of freestanding structures during sintering reduces sagging. Outer structures can be directly supported on the sintering support. Embedded structures have to be stabi-





lized using additional layers like MSP or FSP. Customized sintering profiles should then be applied. Customized sintering profiles for integrated thick-

film metallization reduce warping.

### 3.2 Structural elements for reinforcement

Some devices require deforming bodies with reinforced structures to supply flexural resistant properties (e.g. accelerometers or force sensors). For a precise and reproducible fabrication special techniques were established to process these bodies in multilayer technology:

- Little bars fix the reinforced regions to the outer frame during stacking and laminating (Figure 11).
   After laminating or sintering, these bars have to be removed by laser cutting. However, it must be considered that no other functional structures are placed under the bars which might be damaged by the laser.
- Reinforcing layers, e.g. reinforced centers of diaphragms, can also be stacked separately, as shown in Figure 11. However, this technique requires a customized stacking tool and the process gets more complex.





### 3.3 Dimensional tolerances





Design miniaturization together with panel production of LTCC microsystems offers low-cost applications. This is one of the advantages of the LTCC technology.

However, small dimensional tolerances have to be met in order to provide reproducible properties of the sensors and to achieve an acceptable yield:

- Low tolerances have to be ensured for the structuring processes (punching, laser structuring, screen printing and stacking) over the whole working space of a multiple printed panel.
- The lamination process has to be controlled (e.g. plan parallelism at the uniaxial lamination press) to ensure homogeneous results.
- The nominal shrinkage varies over the fired substrate up to 0.5% (Figure 12). It depends on the lamination process, the structures [19] and the sintering process. For best results, the variation of the shrinkage over the working space of the panel has already to be considered in the CAD files during the design phase.

### 4. Sensor design

Even though the LTCC technology provides many different applications for mechanical sensors, the general design process is almost the same (Figure 13).



**Figure 13:** General design process for mechanical sensors in LTCC.

At the beginning, technical specifications have to be defined (e.g. sensor properties, dimensions and boundary conditions).

Following that, the conceptual design must be specified regarding producibility of structures and useability of materials. After evaluation and selection, the most suitable conceptual design has to be designed in detail.

Today, model-based design and design optimization is inevitable when designing integrated sensors for mechanical quantities. Therefore, physical models of adequate levels of abstraction (granularity) have to be provided for the conceptual design and the final design step as well.

Afterwards, the final design has to be checked and revised if necessary regarding the required properties. For this purpose, an evaluation step follows which includes fabrication and characterization of prototypes. In case of unsatisfying properties, one or more development loops have to be passed to adapt sensor concept and design and to validate or improve the simulation models as well.

Technology-inherent distributions of dimensions, material properties and process parameters lead to performance variations of a set of sensors even when they all are of the same design and fabrication processes. Performance distributions can be minimized by a further design step which involves the probability distributions of the design and process parameters.

The objective of a so-called robust design optimiziation is to find a design that fulfills the target requirements specified with minimized scattering of the sensor performance [20, 21]. As a result, an improved yield is to be expected.

### 5. Applications

### 5.1 Pressure Sensor

Integrated LTCC-based pressure sensors have many advantages in comparison to classic steel or ceramicbased pressure sensors [22-24] because of the technology-inherent features.

The LTCC technology enables easily variable sensor geometries e.g. different diaphragm thicknesses for different pressure ranges by using different tape thicknesses. All types of pressure sensors (relative, absolute, differential) can be built up. Furthermore, all components of the sensor system (sensor body, pressure conSymmetrical axis

3

nectors/micro piping, and electronic components) can be integrated in the LTCC-based multilayer component.

**Figure 14:** Finite elements analyses of different constructional designs (numbers of fixation cantilever): perfect mechanical decoupling of the sensor cell with one or two fixation cantilevers.

In [23] a new LTCC-based pressure sensor concept was presented aiming the improved mechanical decoupling of the diaphragm. The sensor cell is fixed by thin LTCC cantilevers containing micro channels for the pressure connection of the sensor cell.

Using the piezo-resistive measuring principle the strain caused by diaphragm deflection is measured by thickfilm resistors, connected to a Wheatstone bridge and screen printed at the surface of the LTCC diaphragm.

FE analyses were carried out for the optimization of the design of the LTCC fixation cantilever [Figure 4].



**Figure 15:** Characteristic curves of different sensors (pressure ranges). Shift of bridge voltage  $(\Delta U_b)$  vs. applied pressure (p).

The sensor characterization showed that all sensors have a strongly linear behavior (bridge voltage vs. pressure) and are nearly free of hysteresis (Figure 15).

Table 2 shows the overall characteristics of the sensors for different pressure ranges (0.2, 1.5, 5 bar nominal pressure). The operation pressure of the different sensors was defined by additional burst tests. All sensors have a 4-times overload safety.

**Table 2:** Characteristics of different sensor types according to DIN/ISO 16086 (offset voltage < 25 mV/V, bridge resistance ~ 25 kOhm, T=25 °C). D – diaphragm diameter, d – diaphragm thickness,  $p_{op}$  – operation pressure, S – sensitivity, L – linearity, H – hysteresis; FS – full scale.

Diaph	ragm	р <sub>ор</sub>	S	L	Н
D [mm]	d [µm]	[bar]	[mV/ V×bar]	[%FS]	[%FS]
4.5	220	5.0	0.4	0.06	0.05
4.5	150	1.5	1.1	0.04	0.01
4.5	45	0.2	4.6	0.07	0.08

Finally, sensors with integrated signal conditioning electronics (amplifier, temperature compensation) were fabricated (Figure 16).



**Figure 16:** LTCC pressure sensor with integrated electronics for signal condition.

### 5.2 Force Sensor

Force sensors always detect the effect (deformation, stress, strain) of an applied force on a deformable body. The deformation is converted into an electrical signal by transducers working on the principle of e.g. capacitive, piezoelectric or piezo-resistive measurements. Though, the piezo-resistive principle is best suited because of its high accuracy, long-term stability and application range. When this transducer principle is applied, cantilevers and diaphragms obtain the best performance for small nominal loads.



Figure 17: The cartwheel structure [8]. C - cantilever; Z - flexural resistant center; P – piezo-resistors; F - frame.

Miniaturized piezo-resistive force sensors for tensile and compressive loads (2 N, 5 N and 10 N) were developed. Compared to the pressure sensor above, the uniform application of the force to be measured in the deforming sensing body is the main challenge of this study. For this purpose, a cartwheel structure was designed (Figure 17). It consists of four identical cantilevers which are combined by a flexural resistant center. A metal pin with thread is mounted axially in the center hole. This structure obtains a uniform distribution of the applied tensile or compressive forces on the cantilevers, avoids angle errors and combines both, the high sensitivity of cantilevers and the robustness against shear forces of diaphragms.

Four piezo-resistors are placed on the cantilevers in areas of maximum strain – two under expansion and two under compression. They are connected to a Wheatstone bridge, thus the bridge voltage composes the sensing signal.



**Figure 18:** LTCC force sensors for three nominal loads (2 N, 5 N, 10 N). Dimensions: 14 x 14 mm<sup>2</sup>.

An analytical model [8] was developed for the dimensioning and simulation of the sensor elements. The most important restrictions for the design process were (i) to achieve a high grade of miniaturization, (ii) to provide robustness against overloads up to 200% of the nominal load and (iii) an equal layout for the mentioned force ranges except the top layer (Figure 18).

**Table 3:** Measured sensor characteristics (T =  $25^{\circ}$ C). F<sub>B</sub> – breaking load, S – sensitivity, L – linearity, TC-S – temperature coefficient sensitivity, FS – full scale.

Nominal load F N	F <sub>β</sub> [N]	S [mV/( V·N)]	L [%FS]	TC-S [%S/K]
2 N	4	2.6	< 0.6	0.02
5 N	> 10	0.6	< 0.4	0.03
10 N	> 20	0.1	< 1.0	0.02

The designed sensors were fabricated in multiple processing with 25 elements per 4-inch substrate. The techniques: pre-lamination of all sheets, uniaxial cold lamination at 30°C and the fixation of the deformable bodies on the sinter support were used to minimize sagging.

The measured characteristics in Table 3 show the potential of the LTCC as base material for this application. The sensors have a linear behavior and a low temperature drift of the sensitivity. Furthermore, there is a good compromise between sensitivity and breaking load.



**Figure 19:** Characteristic curves of different sensors (load ranges). Shift of bridge voltage ( $\Delta$ Ub) vs. applied force (F).

### 5.3 Acceleration Sensor

The measurement of accelerations requires a mass element on a spring. The mass elongates and deforms the spring in response to an applied acceleration. The deformation can be measured e.g. by a piezo-resistive transducer. Today's acceleration sensors made of silicon offer sufficient functionality in a cost-effective way. In contrast, acceleration sensors made of LTCC work under elevated temperatures, or as an additional feature integrated in LTCC substrates or electronic assemblies.

The LTCC multilayer technology suggests a sensor layout with leaf springs in a layer stressed for bending or for torsion. Thus, an acceleration perpendicular to the layer effects a deformation of the springs which can be measured by piezo-resistive thick-film resistors printed on the springs. The layout has to consider some conflicting requirements, e.g. high resonance frequency, i.e. high stiffness versus high sensitivity, i.e. lower stiffness, or uniform strain in the benders for a high reliability versus high strained areas of the springs for a high sensitivity.

Analytical and finite element models were used to optimize the sensor design. Two parallel trapezoidal benders have advantages as compared to rectangular benders or torsion springs, or the bridge and cantilever structures proposed in [25]. A FEA model of the optimized layout fabricated in LTCC is shown in Figure 20.



**Figure 20:** FEA model of an acceleration sensor with two trapezoidal benders (equivalent strain).

The first resonance frequency has a strong influence to the working range and is obtained from FEA models. A linear relation between acceleration and deformation can be expected about 50% of the resonant frequency. Resonant excitation would destroy the sensor. Four thick-film resistors, two on the benders and two on the frame are connected to a Wheatstone bridge like it has been done in case of the pressure sensors.



**Figure 21:** LTCC acceleration sensor with piezo-resistors on rectangle leaf springs.



**Figure 22:** LTCC acceleration sensor characteristic curve, bridge voltage  $(U_b)$  vs. acceleration. Different excitation frequencies.

Figure 21 shows a LTCC acceleration sensor with a resonance frequency of 250 Hz [20]. Excited with 100 Hz the sensor gives a slightly higher signal due to the closer proximity to the resonance frequency as compared to 50 or 75 Hz (Figure 22).

However, the tested sensors are sensitive for large accelerations. Smaller effective ranges or can be designed.

### 5.4 Flow Sensor

Most of the commercial flow sensors work on the heating wire principle. A thin platinum wire which is heated by a current is placed in a flowing gas. It cools the platinum wire which refers to its resistance and influences the connected Wheatstone bridge. The implementation of a channel system with integrated freestanding heating wire in LTCC is only possible when using a sacrificial material. A thin heater can be realized by overprinting the FSP with a thick-film platinum paste. It enables low power loss and raises the sensitivity. In order to reach an adjusted shrinkage between LTCC and FSP a zero-shrinkage technique is required. For this purpose, several techniques were investigated:

- The combination of DP 951 with Release-tapes (Ceramtape A, GT 951 RT) indicates delamination and residues at removal respectively.
- The self-constrained LTCC-tape HL2000 (Heraeus) is not suitable to uniaxial lamination process (voids and delamination) which is required and the FSP-paste (residues after firing).
- Best results were achieved with a combination of DP 951 and HL2000 (Figure 23). DP 951 suits to the FSP-paste and stabilizes the HL2000 during the lamination process. The zero shrinkage of the whole substrate is constrained by the HL2000.





Figure 23 illustrates the final sensor design [Loh12]. The bottom part of the channel was filled with sacrificial material, which was overprinted by glass and platinum paste to create the heater structure.

Afterwards, the upper channel part was laminated on top of it so that the heater was centered. With this technique a lot of different heater geometries can be realized.

**Table 4:** Sensor characteristics.  $d_{H}$  – heater thickness,  $L_{H}$  – heater length,  $R_{H0}$  – heater resistance (T = 25 °C),  $R_{H100}$  – heater resistance (T = 100 °C).

Channel cross section A <sub>c</sub>	d <sub>н</sub> [μm]	L <sub>H</sub> [mm]	R <sub>Η0</sub> [Ω]	R <sub>Η100</sub> [Ω]
0.8 mm	50	4	2.5	3.1

The prototypes show the expected relation between the current through the heating wire and the fluid flow. Figure 24 shows the characteristic curve of the sensor. In future work, a measuring circuit will be integrated in the system to complete the sensor and to linearize the current-flow correlation.





Fabricated flow sensor with assembled fluidic adapters

CT-scan of the fabricated flow sensor



Figure 24: LTCC flow sensor and characteristic curve, heater current (I) vs. fluid flow (v).

### 6. Conclusions

The selected examples show that LTCC is a well suited integration platform for mechanical sensors. Their design and fabrication requires a deep understanding and control of material issues (e.g. material interactions during co-firing, shrinkage behavior, sacrificial layer). Besides this, it is necessary to expand the LTCC process flow on the generation of the required 3D structural elements.

Pressure, force, acceleration and flow sensors were developed following a defined design and manufacturing flow.

It was shown that the sensors have an excellent and stable functionality in all cases. The main advantage of the presented sensors is the quasi-monolithic design. It prevents thermo-mechanical strain which normally occurs when parts of different materials are bonded. Using the demonstrated material and process expansions and improvements, LTCC-based sensors for mechanical quantities are interesting and cost-effective alternatives to existing technical solutions.

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# Wireless LTCC sensors for monitoring of pressure, temperature and moisture

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**Abstract:** In this paper five different wireless sensors operating in the MHz range are presented. Embedded pressure sensors, temperature sensor and sensor for moisture detection all based on a passive wireless inductor-capacitor resonant circuit are realized, following specifications of the LTCC technology design and fabrication process. Considered resonant sensor principle is based on the passive resonant circuit (LC circuit), where changes are detected by variation of sensor capacitance, while inductor inductance remains invariable.

Key words: pressure sensors, temperature sensor, sensor for moisture detection, LTCC.

### Brezžični LTCC senzorji za merjenje pritiska, temperature in vlage

**Povzetek:** V članku je predstavljenih pet različnih brezžičnih senzorjev, ki delujejo v MHz področju. Vgrajeni senzorji pritiska, temperature in vlage temeljijo na brezžičnem induktivno-kapacitivnem resonančnem vezju in so izvedeni po specifikacijah LTCC tehnologije in proizvodnega procesa. Način pasivnega LC resonančnega vezja deluje na spremembo kapacitivnosti, pri čemer induktivnost ostaja nespremenjena.

Ključne besede: senzorji tlaka, senzorji temperature, senzorji vlage, LTCC.

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### 1. Introduction

Monitoring of physical parameters, such as a pressure, temperature, moisture etc. and its measurement are widely employed in different areas of everyday life [1-5]. Precise detection of pressure or temperature variations is important in many applications e.g. automotive or aerospace industry, internal combustion or turbine engines etc. Control of moisture in products can be a vital part of the process of the product. The measurement of moisture has been of interest to building professionals for many years. Development of innovative methods for monitoring and measuring physical parameters in the civil engineering is therefore both commercially and scientifically important.

Sensors should have high sensitivity for the measured parameters and they should be insensitive to the other parameters of environment. Sensitivity should be followed with linearity of measured values in order to simplify the sensing element configuration. Small dimension of the sensors, low cost and non-contact measurement system for data retrieval are very often required.

Choice of the adequate technology and properties of materials involved in the sensor realizations can ensure its application in chemically aggressive environments and in environments with extreme operating conditions. Since devices fabricated in the Low Temperature Co-fired Ceramic technology (LTCC) are based on glass ceramics they are very well suited for harsh environments. LTCC has been proven as a valuable tool for the realization of three dimensional microsystem structures [4-9]. The variety of available LTCC tapes, as well as a number of methods for substrate pattering, possibility of integration in one LTCC module fluidic channels, heaters, sensors, electronics put LTCC technology in the high-ranking place among technologies suitable for the fabrication of compact sensors devices.

This paper proposes a realization concept of a five resonant sensors configurations. Embedded pressure

sensors, temperature sensor and sensor for moisture detection all based on a passive wireless inductor-ca-pacitor resonant circuit are realized.

In section 2 are presented design, detection principle, fabrication and measured results for pressure sensors. Temperature sensor and sensor for detection of moisture in building materials are presented in Section 3 and Section 4, while in section 5 conclusions are made.

### 2. Pressure sensors

Three pressure sensors are designed and fabricated in the LTCC technology. The sensors are realized as a parallel resonant circuit where pressures changing are detected by variation of capacitance, while inductor remains constant.

### 2.1 Design, geometrical parameters and theoretical model of the pressure sensors

The first sensor can be found suitable for applications in chemically aggressive environments, since sensor membranes are formed as a sandwich composition comprising electrode layers placed between two dielectric tapes and connected in parallel with inductor windings, Fig. 1. The sensor is built up by seven laser



D) Closs section

Figure 1: Pressure sensor type 1.

micromachining tapes carrying out vias and holes. Capacitor plates are printed on the top of layer 5 and to the bottom of layer 3, while inductor is placed on the layer 6. Capacitor electrodes are embedded in an each of membranes, which are separated by the cavity. Membranes exposed to the pressure to be measured are formed by the tape layer 2, 3, 5 and 6.

The second design of the pressure sensor incorporates capacitor electrodes that are placed on the outer side of membranes enabling direct application of the pressure to be measured, Fig. 2. Formation of the outer electrodes is attained by implementation of the thin film metallization deposited in the post firing process. This procedure for sensor realization differs from the firstly presented sensor configuration with buried electrodes, resulting in increased sensitivity.

The last sensor design of the pressure sensor was realized with intention to minimize overall sensor dimension. The inductor coil is realized in two layers resulting in miniaturization of the sensor overall dimension, Fig. 3.





Measured relevant electrical and mechanical characteristic of used LTCC tapes are presented in the Table 1 and Table 2, while geometrical parameters of all sensors are presented in the Table 3 and Table 4.



Figure 3: Explode view of sensor type 3.

Table 1: Physical properties of LTCC tapes.

Material	Thickness in green state	Thickness after firing
CeramTec GC	100 [μm]	80 [µm]
Heraeus CT707	125 [μm]	95 [μm]

Table 2: Measured parameters of LTCC tapes.

Material	Relative permittivity @ 1 kHz	Young's modulus @ 25 °C
CeramTec GC	7.8	61.36 [GPa]
Heraeus CT707	6.39	53.49 [GPa]

**Table 3:** Geometrical parameters of inductors design.

Parameters	Dimension		
	Type 1	Type 2	Type 3
din	10 [mm]	10 [mm]	10 [mm]
S	200 [µm]	270 [µm]	270 [µm]
W	350 [µm]	285 [µm]	285 [µm]
N	17.5	17.5	9.75 (x2)

 $d_{in}$  - minimal distance between opposite segments of inner winding, *s* - spacing between adjacent segments, *w* - width of conductor line, *N* - number of windings.

Table 4: Geometrical parameters of capacitors design.

Parameters	Dimension		
	Type 1	Type 2 and Type 3	
av	3.3 [mm]	3.5 [mm]	
а	4 [mm]	4.35 [mm]	
tcond	12 [µm]	12 [µm]	
tm	80 [µm]	95 [μm]	
tg	80 [µm]	95 [μm]	

 $a_v$  - hole radius, a - electrode radius,  $t_{cond}$  - conductor thickness,  $t_m$  - tape thickness,  $t_a$  - cavity thickness.

Overall dimensions of the fabricated sensors are presented in Table 5.

Table 5: Overall sensors dimension.

Sensor	Dimension
Type 1	35 x 29 x 0.56 [mm]
Type 2	35 x 29 x 0.665 [mm]
Type 3	24.5 x 20.5 x 0.665 [mm]

Theoretical model and wireless detection will be explained for the sensor type 2. Pressure variations are detected by changes in the sensors capacitance during deflection. The sensor capacitance is a complex structure composed from four capacitances - two directly under/above the air-gap ( $C_1$  and  $C_3$ ), the air-gap capacitance ( $C_2$ ) and the annulus capacitance ( $C_4$ ), Fig. 4.



**Figure 4:** Sensor capacitance corresponding to attribution of individual capacitances.

Deflection of membranes occurs when pressure is induced, resulting in the change of the sensor overall capacitance,  $C_s(P)$ . Value of  $C_s(P)$  has been derived in [8, 9] and can be obtained as

$$C_{s}(P) = C_{s}(0) \frac{ar \tanh\left(\sqrt{\frac{2d_{0}(P)}{t_{g} + \frac{2t_{m}}{\varepsilon_{r}}}}\right)}{\frac{2d_{0}(P)}{t_{g} + \frac{2t_{m}}{\varepsilon_{r}}}} + \frac{\varepsilon_{0}\varepsilon_{r}\pi\left(a^{2} - a_{v}^{2}\right)}{2t_{m} + t_{g}},$$
(1)

where  $C_{s}(0)$  is the equivalent capacitance for the sensors at zero pressure can be determined as,

$$C_{s}(0) == \frac{\varepsilon_{0}\varepsilon_{r}a_{v}^{2}\pi}{2t_{m} + \varepsilon_{r} \cdot t_{g}} + \frac{\varepsilon_{0}\varepsilon_{r}\pi \cdot \left(a^{2} - a_{v}^{2}\right)}{2t_{m} + t_{g}}.$$
(2)

and  $d_{0}(\mathbf{P})$  represent the central deflection of membranes.

Values of the membranes central deflection increase under load consequently leading to increase the overall capacitance of the sensor as can been seen from the Fig 5.



Figure 5: Sensor capacitance versus applied pressure.

For the determination of the inductance values monomial expression present in [10] is used.

Sensor resonant nature enables a wireless system for data retrieval using antenna coil, which is present on the Fig 6.



Figure 6: Wireless measurement setup scheme.

Resonant frequency of the sensor antenna system is a pressure dependent value and can be determinate from the system input impedance when the imaginary part of that value becomes zero, Fig 7. Dependence of the resonant frequency of the sensor antenna system versus pressure are presented on the Fig. 8 and Fig. 9.



**Figure 7:** Imaginary part of the input system impedance versus frequency for zero pressure.



**Figure 8:** Imaginary part of input impedance versus frequency for different pressure values.



Figure 9: Resonant frequency versus pressure

As can been seen from the Fig. 8 and Fig. 9 that pressure variations influence on the capacitance value, resulting in a shift of the sensors resonant frequency. Increase of the pressure induced onto sensor membranes leads to an increase of the sensor capacitance and consequently results in a decrease of the system resonant frequency value.

#### 2.2 Fabrication and measured results

Sensor type 1 is fabricated by application of the standard LTCC technology which covers structuring of tapes using laser micromachining, metalizing, laminating, and finally co-firing the stack of LTCC tape layers. CeramTec GC tapes and compatible silver pastes (Heraeus TC 7303 for line printing and Heraeus TC 7304 for via filling) [11, 12] have been used for the sensor fabrication. Isostatic lamination of the collated LTCC layers has been performed at pressure of 50 bar, temperature of 75 °C, and exposure time of 5 min. Firing of the laminated LTCC stack has been conducted in a six zone belt furnace at peak temperature of 900 °C and total firing cycle time of 210 min.

Sensors type 2 and type 3 is also realized implementing the conventional LTCC technology. Heraeus CT 707 tapes are combined with compatible silver pastes (Heraeus TC 7303A for line printing and Heraeus TC 7304 for via filling) [12] are used for sensor fabrication. Collated tape layers are isostatically laminated at pressure of 70 bar and temperature of 75 °C for 5 minutes. The laminated stack is fired in a six zones belt furnace at peak temperature of 880 °C and total firing cycle time of two hours. This is followed by thin film deposition of silver electrodes onto sensor membranes using the sputtering method.

Resonant operating principle of the sensor allows the possibility for the usage of wireless measurement system for the data retrieval. The measurement setup comprises an antenna and a device for the resonance detection. During the measurement procedure, the sensor is placed in the center of the antenna. The test setup for wireless recording of sensor characteristics is built up by a clamping system where two plates made from acrylic glass, are carrying ducts for the supply of pressurized air, Fig. 10. The exhaust holes for the air in the center of the plates are positioned exactly above the sensor membranes exerting the pressure to be measured. The test setup is additionally equipped with a rectangular antenna coil required for the wireless measurement which is connected to the spectrum analyzer (Anritsu MS620J).

The measured results, compared with the theoretical results for the sensor type 2 are presented in the Fig. 11, while compared measurement results of all sensors are presented in the Fig. 12.



Figure 10: Test setup for the measurement.



**Figure 11:** The theoretically and measured results for the pressure sensor type 2.



**Figure 12:** Relative change of sensors resonant frequency versus pressure (measured results).

As can been seen from the Fig. 11 there is a good agreement between theorethical and measured results. It is proved that theorethical model describe sensor anntena system very well. From the Fig. 12 can be seen that formation of the sputtered thin film electrodes and usage of tapes with higher elasticity significantly contributes to the increase of sensor sensitivity compared to designs for sensor type 1. In addition, 3D realization of inductor coil does not decrease the sensor sensitivity, while the overall dimension of sensor can be significantly reduced.

### 3. Temperature sensor

Temperature measurement is state of the art using many different techniques reaching from thermocouples over resistors (PTC, NTC, Pt100) to fully digital wireless silicon systems. The approach to use a temperature dependent capacitor along a coil to form a resonating structure leads to a wireless temperature sensor that is embedded in a rugged ceramic body and resistant to corrosive environments and can be read by means of an antenna coil.

The sensor introduced in this paper permits temperature measurement in a high pressure, high temperature and even corrosive environment due to its realization in LTCC technology. Design of the temperature sensor is presented on the Fig. 13.



Figure 13: Temperature sensor exploded 3D view.

The sensor consists of several layers of LTCC tapes where the inner layers carry the actual sensor components (inductor and capacitor) and additional outer layers increase the mechanical stability of the device. Electrodes of the capacitor are screen-printed on the top of layer 2 and bottom of layer 6, while the square spiral shaped inductor is place on top of layer 6. For the realization of the sensors, Heraeus CT707 in combination with Heraeus CT765 (sensitive dielectric - ferroelectric) are used. The electrode radius is 4 mm, while geometrical parameters of inductor are presented in the Table 6.

Parameters	Dimension
din	10 [mm]
S	270 [µm]
W	285 [µm]

17.5

Table 6: Geometrical parameters of inductors design.

 $d_{in}$  minimal distance between opposite segments of inner winding, *s* - spacing between adjacent segments, *w* - width of conductor line (*w*), *N* number of windings.

Ν

The working principle of the sensor is based on a resonant circuit (inductor and capacitor) and changes its resonant frequency due to thermal expansion of the bulk material and change in permittivity of a special dielectric layer. The capacitance of sensor can be calculate using the following equation [13].

$$C_s = \frac{\varepsilon_0 \cdot \varepsilon_r(T) \cdot A(T)}{d(T)} \tag{3}$$

where A(T) is temperature dependent plate area d(T) temperature dependent plate distance and  $\varepsilon_{re}(T)$  temperature dependent relative permittivity.

The biggest influence on changing the sensor capacitance with the temperature has relative permittivity of the material. Relative permittivity increases if ambient temperature raises leading to increasing the capacitance of the sensor, while value of inductance remains constant, as a resulting that sensor resonant frequency decrease.

The sensor is fabricated using the standard LTCC fabrication process. Tape layers were first structured with a Nd:YAG laser. After the following screen-printing step using Heraeus TC7304 as via-filler and TC7303 as conductor paste, the layers are dried, stacked in a lamination fixture and laminated for 3 minutes at a temperature of 70 °C and a pressure 60 bar in an isostatic press. Firing of the tapes has been performed in a conventional six zones thick film furnace with a cycle time of 2 h and 880 °C peak temperature. Fabricated sensor element is presented on the Fig. 14.

The measurement set-up shown in Fig. 15 consists of a heat resistant alumina plate that is held 2 cm above a jig that contains a rectangular antenna coil Cu-wire. A hot air stream heats the sensor that is located on the top of the alumina plate and is surrounded by heat resistant insulating bricks (the bricks have been removed for clarity in Fig. 15) to prevent excessive heat loss. Temperature measurement via thermocouples on top and bottom side of the sensor provided defined thermal conditions during measurement.



Figure 14: Fabricated temperature sensor (top view).



Figure 15: Measurement set-up.

For the measurement, a network spectrum analyzer is connected to the antenna coil via two BNC-terminated coax cables (RG58). Measurements are taken as linear magnitude plots in the relevant range. The measured results for the temperature sensor is presented in the Fig. 16.



Figure 16: Resonant frequency versus temperature.

As can been seen from the measured results that the resonant frequency of the sensor decreasing when ambient temperature increase. Reason for that is the capacitance of the sensor which increases due to the increasing of the relative permittivity of material over the temperature.

## 4. sensor for detection of moisture in building materials

In order to know the condition of buildings and other construction structures in the construction industry, there is a need to monitor moisture content. In this way, small (and cheap) repairs at the right time, to extend the life time of a building, are ensured. This is especially important for objects which are sensitive to environmental influences.

In this section it will be presents a LTCC sensor for measuring moisture content of building material (clay brick). The proposed sensor consists of two dielectric layers [14], Fig. 17. The LC structure was screen-printed on the first dielectric layer (as a substrate) and the second layer has a window over capacitor's electrodes. Through this window the sensor is exposed to moisture (thanks to the hydrophilic behaviour), which will then cause change of its relative permittivity and total capacitance, and consequently the resonant frequency of the LC sensor.



**Figure 17:** Exploded view of the sensor for moisture detection.

This LC sensor is realized that the inductance of the sensor remains constant. The inductive part was covered
with dielectric layer. Contrary to this, the capacitance of the interdigitated electrode system is changed with the variation of the permittivity of the medium (exposed through the small window from the top side of the sensor). This will cause the shift of sensor's resonant frequency. The capacitance of the interdigitated can be calculate using equation proposed in [15, 16],

$$C_s = \frac{\varepsilon_{re} \cdot 10^{-2}}{18\pi} \cdot \frac{K(k)}{K'(k)}$$
(4)

where *I* is length of the fingers expressed in micrometers and *N* is number of fingers. The ratio of complete elliptic integral of first kind *K*(*k*) and its complement *K*'(*k*) is given by [4,5].  $\varepsilon_{re}$  is the effective dielectric constant of the microstrip line width *w*, and define by following equation,

$$\varepsilon_{re} = \frac{\varepsilon_r + 1}{2} + \frac{\varepsilon_r - 1}{2} \cdot F\left(\frac{w}{h}\right) - M \tag{5}$$

F and M are given explain in [15, 16].

As can been seen from equation (4) and (5) if relative permittivity is higher capacitance of the sensor will be bigger, while inductor remains constant, resulting in a decreasing the resonant frequency of the sensor.

Dimensions of the sensor elements are presented in the Table 7 and Table 8.

Table 7: Geometrical parameters of inductors design.

Parameters	Sensor
din	7.6 [mm]
S	100 [μm]
W	500 [μm]
N	13

 $d_{in}$  - minimal distance between opposite segments of inner winding, *s*- spacing between adjacent segments, *w*- width of conductor line, *N* - number of windings.

Table 8: Geometrical parameters of capacitor design.

Parameter	Dimension
W	500 [μm]
S	100 [μm]
	13.2 [mm]
N	18
dx	13.2 [mm]
dy	21 [mm]

*w* - finger width, *s* - spacing, *l* - length of fingers, *N* - number of fingers,  $d_{y}$  - total length,  $d_{y}$  - total width

Total dimension of the sensor are (38.2 x 24 x 0.4) mm.

The sensor was fabricated using the LTCC technology, comprising the processing steps of structuring tapes by means of laser micro-machining, metallizing, laminating and co-firing the stack of two LTCC tape layers. For the sensor fabrication Heraeus CT700 [12] dielectric tapes were combined with compatible silver paste Heraeus TC 7303A for conductive line printing. Collated tape layers were isostatically laminated at pressure of 70 bars and temperature of 75 °C for 3 minutes. The laminated stack was fired in a six zones belt furnace at a temperature peak of 880 °C and a total firing cycle time of 2 hours. Fig. 18 illustrates the fabricated sensor. In the Fig. 19 is presented measurement setup and measured results (for the clay brick) are presented in the Fig. 20.



**Figure 18:** Fabricated sensor for moisture detection (top view).



**Figure 19:** Experimental setup for measuring the resonant frequency of the sensor embedded into building material.



Figure 20: Resonant frequency versus percent of water absorption.

The measure results shows that the resonant frequency changes from 109.2 MHz to 106.9 MHz while the percentage of water ranging from 2% to 17.5%. The variation of resonant frequency changes is 2.3 MHz while the change in moisture content is 15.5%. It can also be seen that the deviation of measured values from the ideal linear dependence is very small. The sensitivity of the sensor in this case is 169 kHz/percentage of water content.

## 5. Conclusion

The sensor modules presented in this paper are a passive LC resonant type sensor designed for the operation in MHz range and are fabricated using the conventional LTCC technological process. Three pressure sensors, one temperature and one sensor for the moisture detection are designed and fabricated in the LTCC technology. For all sensors a wireless measurement set-up is used for non-contact retrieval of the measured data.

It is shown that formation of the sputtered thin film electrodes and usage of tapes with higher elasticity significantly contributes to the increase of sensitivity of the pressure sensor. While 3D realization of inductor coil does not decrease the sensor sensitivity, it contributes to the significant reduction of sensor overall dimensions.

The experiments have shown, that LTCC technology offers the possibility to realize different wireless readout sensors with nearly linear characteristics.

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## Wide Band-stop Microwave Microstrip Filter on High-resistivity Silicon

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**Abstract:** Low-resistivity silicon (standard CMOS-grade silicon) is a troublesome issue for the microwave passive components. One solution is using high-resistivity silicon (HRS). Wide band-stop filter on high-resistivity silicon (HRS), 5 k $\Omega$ -cm, is introduced. The filter is simulated and successfully fabricated in microstrip technology. Relative stop band width is over 70 % for 20 dB suppression. Reflection, S11, in the pass band is -17 dB or lower. Metallization was sputterd aluminum (AI) and silver epoxy was used for bonding.

Key words: Microwave, High-resistivity silicon (HRS), Band-stop filter, Sputtered aluminum (Al)

# Širokopasovni zaporni mikrovalovni mikrotrakasti filter na visoko uporovnem siliciju

**Povzetek:** Nizko uporovni silicij (standardni CMOS silicij) ni primeren za mikravalovne pasivne elemente. Ena izmed rešitev je visoko uporovni silicij (HRS). Predstavljen je širokopasoven zapeerni filter na visoko uporovnem 5 kΩ-cm siliciju. Filter je simuliran in uspešno izveden v mikrotrakasti tehnologiji. Relativna bločna širina je preko 70 % pri dušenju 20 dB. Refleksija S11 v prepustnem pasu je -17 dB ali manj. Metalizacija je izvedena z naprševanjem aluminija, bondiranje pa s srebrno pasto.

Ključne besede: mikro valovi, visoko uporovni silicij (HRS), pasovni zaporni filter, napršeni aluminij

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## 1. introduction

Microwave passive components on silicon (Si) substrate in microstrip technique are important for microwave integration. Wide band-stop filters are, also, important function of passive components. Si is a vital material for present microelectronics but has some functional problems.

Low-resistivity silicon (standard CMOS-grade silicon, resistivity 1 - 30  $\Omega$ -cm) is a troublesome issue for the microwave passive components [1], [2]. Due to high substrate loss it is a problem to fabricate useful traditional passive microwave components on such substrate. One of the solutions is to apply benzocyclobutene (BCB) to serve as the interface layer [2], [3]. BCB has a low dielectric loss and low dielectric constant. The problem is a new material in technology and a weak compactness of the structure due to two substrates. Thin BCB layer, also, need very narrow microstrip lines (<< 100  $\mu$ m).

Another solution is using high-resistivity silicon (HRS) with resistivity higher than 2 k $\Omega$ -cm [1], [4]-[7]. Simulated and successfully fabricated microstrip attenuator

on high-resistivity silicon (HRS) was presented in [4]. In [4] microstrip lines were fabricated as a metallization of 2 µm thick sputtered aluminum (Al) layer. Examples of the sputtered Al applied to HRS was, also, reported in [5,6]. Aluminum was chosen as the conductor because this is the standard foundry metal [6], even for the band around 30 GHz [5].

The aim of this paper is to simulate and fabricate wide band-stop filter on the HRS substrate. The idea is to simulate circuit with ideal waveguides and to apply it for fabrication of microstrip filter structures [8]. The solution is adjusted to the conditions of HRS and common etching technological ability. The narrowist microstrip line is fixed to 50 µm for the classical etching technology. Metalization is sputtered Al and silver epoxy is used for bonding SMA connectors to the microstrip lines [9, 10].

## 2. Design and simulation

Calculation of the circuit with the ideal waveguide is done according to design curve presented in [8]. The



**Figure 1:** Ideal circuit of the introduced low-pass filter,  $Z_2 = Z_{s1} = 84.2 \Omega$ ;  $Z_1 = Z_{s2} = 31.9 \Omega$ 



Figure 2: Ideal microstrip presentation of the filter (narrow line, 84.2  $\Omega$ , is 50  $\mu$ m).

curves present optimal values of the pair of maximum and minimum characteristic impedances.

Characteristics of the used HRS substrate are: substrate height  $h = 300 \ \mu$ m, relative dielectric constant  $\varepsilon_r = 11.7$  and resistivity is  $\rho = 5 \ k\Omega$ -cm. Dielectric losses were chosen to be tg $\delta = 0.02$ . Metallization is Al thickness  $t = 3 \ \mu$ m with chosen conductivity to be low, 6 MS/m. The highest characteristic impedance is to adjust common etching technological ability. It is related to the minimum microstrip line width which is chosen to be 50  $\mu$ m and corresponds to 84.2  $\Omega$ . The lower characteristic impedance is 31.9  $\Omega$ . Ideal filter circuit and the ideal filter in microstrip technology are presented in fig. 1 and fig. 2, respectively.

Fig. 3 presents pattern of the introduced filter. Wide microstrip lines are something shortened according to influence of edge effect. Open stubs are folded to reduce the size of the structure. 50  $\Omega$  lines (240  $\mu$ m wide) for connectors are included at both ends, as shown in fig. 3.



**Figure 3:** Compact version of the filter (19.22 mm x 11.36 mm). Narrow lines are 50  $\mu$ m wide ; wide lines are 560  $\mu$ m wide.

Simulation results for the ideal model, fig. 1, and 3D electromagnetic simulation (IE3D Zeland Software ver. 10) of the real pattern, fig.3, are presented in fig. 4. They

are in agreement for frequency position of the stopband minimum and the filter shape. Logical difference is for losses, especially in the pass-band region. Relative band-width of the stop band is around 70% for 20 dB suppression.



Figure 4: S<sub>21</sub> of ideal circuit and 3D EM simulation

#### 3. Fabrication and measurement

Photo of the fabricated filter with SMA connectors is presented in fig. 5. Aluminum (Al) with 1% of Si was sputtered on Si substrate (wafer) to form metallization 3 µm thick. Content of 1% of Si obtains no penetration of Al into the Si substrate. Pattern of the filter, presented in fig. 3, was etched in Al metallization. SMA connectors were bonded on the ends of the 50  $\Omega$  microstrip lines (240 µm wide, fig. 3) using silver epoxy. Ground plane is a brass metal plate bonded to the lower side of the silicon substrate using silver epoxy.



**Figure 5:** Photo of the fabricated filter with SMA connector

It was mesured in dark.  $S_{21}$ -parameters for the structure in fig. 5 are presented in fig. 6. Losses in the pass-band are high (-10 dB). Beside that, they are in an agreement with graphics in fig. 4 for frequency position of the stop-band minimum and the filter shape. The problem were only losses. The reason for the high losses was discovered to be out of date (old) silver epoxy.



**Figure 6:** Measured  $S_{21}$  of the filter using out of date (old) silver epoxy

Then, SMA connectors were removed and both the connectors and the contact aresa on the microstrip were cleaned from the old silver epoxy. The SMA connectors were again bonded using the fresh silver epoxy. It was also mesured in dark. *S*-parameters for the filter with the fresh silver epoxy are presented in fig. 7.



Figure 7: Measured (in dark) S-parameters of the filter using fresh silver epoxy

Measurement under light source illumination can degrade filter characteristics in pass-band. The illumination causes higher losses in the pass-band [11] which is not acceptable for the filter. According to that, operation of the filter is only in dark (protected from light). One example of extreme illumination is presened in fig. 8: filter positioned 1m from 1000 W photo lamp gets totally degraded pass-band. Even small power laser (650



nm, around 1 mW), applied at the end of one microstrip stub, increases losses in the pass-band is near 0.3 dB.

**Figure 8:** Measured *S*-parameters of the filter positioned 1m from 1000 W photo lamp. ( $S_{21}$  at 1.2 GHz is near -21 dB comparing with around -3 dB in dark)

Compared  $S_{21}$  parameters for ideal circuit, EM simulation and measured filter are presented in fig.9. All results are in a good agreement. Relative stop band width is over 70 % for 20 dB suppression. Reflection,  $S_{11}$ , in the pass band is -17 dB or lower.



**Figure 9:** Compared results of  $S_{21}$  for ideal circuit, EM simulated and measured filter

Losses in the pass-band can be caused by: 1) connectors, 2) bonding between connectors and AI metallization using silver epoxy, 3) sputtered AI metallization  $t = 3 \ \mu m$  thick, 4) Si dielectric losses 5) mismatching and 6) radiation. Parameter  $S_{11}$  in fig. 7 is less than -17 in the pass-band and mismatching is not a problem. Simulation of the same filter structure (and the same Si dielectric losses) with copper (Cu) metallization as an excellent conductor are presented in fig. 10. (Cu metallization is rarely used and is electroplated which is different technology). In fig. 10 pass-band losses are small, below 1 dB, for frequencies below 1 GHz. Radiation is not significant. Differences in the case of our fabricated filter are: 1) losses in the silver epoxy bonding between SMA connectors and AI metallization and 2) losses in the sputtered AI metallization and its roughness.



**Figure 10:**  $S_{21}$  of 3D EM simulation with copper (Cu) metallization of various thickness *t* 

Passive intermodulation was measured using signals on 1 GHz and 1.05 GHz with 2 x 4 dBm on input port of the filter. Each signal generator has one circulator for isolation and input into the filter is trough a power divider (from two ports to one). Spectrum analyser is connected to the output of the filter. Intermodulation was not noticed.

#### 4. conclusion

Low-resistivity silicon (standard CMOS-grade silicon) is a troublesome issue for the microwave passive components. One solution is using high-resistivity silicon (HRS). Circuit with ideal waveguides was simulated and successfully applied to band-stop microstrip filter on HRS ( $\rho = 5 \text{ k}\Omega$ -cm). The filter was fabricated using aluminum (Al) sputtered metalization and etching of the filter pattern in Al. SMA connectors are bonded using silver epoxy. The fabrication stressed importance of the quality (fresh) silver epoxy for low losses.

There is a high agreement between ideal circuit simulation, 3D electromagnetic simulation in microstrip technology and measured results. Relative stop band width is over 70 % for 20 dB suppression. Reflection,  $S_{11}$ , in the pass band is -17 dB or lower. It means that the sputtered Al and the silver epoxy can be successfully applied for microwave structures on HRS.

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# In memoriam Marija Kosec

Professor Dr. Marija Kosec passed away after a brief and serious illness on 23 December 2012.



Professor Marija Kosec was a very active and strong supporter of Society for Microelectronics, Electronic Components and Materials MIDEM and its long-term member. In the years 1996-2005 she was the President, and in the periods 1989-1996 and 2005-2011 the Vice-President of the society.

She studied chemical technology at the University of Ljubljana and obtained a PhD degree in chemistry in 1982. Since 1971 she was employed at the Jožef Stefan Institute, and since 2002 she was Head of the Electronic Ceramics Department. In 2007 – 09 she was the President of the Scientific Council of Jožef Stefan Institute.

In the years 2004 – 09 she was leading the Centre of Excellence Materials for electronics of next generations and other emerging technologies and since 2009 the Centre of Excellence Advanced Materials and Technologies for the Future NAMASTE.

Since 1999 she was Professor of Materials Science at the University of Ljubljana and the vice-president of Jožef Stefan Postgraduate School. She was visiting professor at Ecole Polytechnique Fédérale de Lausanne, Switzerland, Shizuoka University, Japan and for short periods at a number of other schools. She was Adjunct Professor at Xi'an Jiaotong University, China.

She was the only female member of Slovenian Academy of Engineering since its establishment in 1995 and in 2005 – 06 its President.

Prof. Kosec supported the European Cooperation in Science and Technology framework COST for many years, actively participating in COST Actions since 1991. Until 2010 she represented Slovenia in the COST Materials Nanosciences Physics Domain Committee (MPNS DC). In 2010 she was nominated MPNS DC expert for Slovenia.

Since 1999 she was the member of the European Liaison Committee of International Microelectronics and Packaging Society and since 2001 the member of the Ferroelectrics Committee at IEEE.

Prof. Kosec was Ambassador of Science of Republic of Slovenia (2003). She was the recipient of the Zois award, the highest national science award in 2006. In 2009 she received Puh recognition for the implementation of research results in industry. In 2010 she received the Ferroelectrics Recognition Award, IEEE Ultrasonics, Ferroelectrics, and Frequency Control Society for her significant contributions to the processing science and technology of ferroelectric powders, bulk ceramics, thin and thick films.

Her research interest was in synthesis and characterization of electronic ceramics, particularly piezoelectrics and ferroelectrics in bulk, thin and thick film form. Her contribution to science was in understanding phenomena of ceramics processing of application-important multi-component systems. Her mission was to merge basic research with applied research and development. She participated, as member or principal investigator, in more than 20 European framework or international projects and she had more than 40 contracts with industry.

She was author or co-author of more than 300 scientific papers in international journals with more than 2000 citations. She gave more than 150 invited talks at international conferences and at different research institutions including Max Planck Institut, MIT, Tokyo Institute of Technology, and at important Japanese producers of electronic components including Murata, TDK, Panasonic and Toshiba.

She chaired the international conference Electroceramics VII in 2000, the European conference Processing of Electroceramics in 2003 and the 4th European Microelectronics and Packaging Symposium in 2006.

In an effort to strengthen the cooperation between science and industry she organized, together with the Technology Centre SEMTO, a series of meetings where researchers, developers and users could present results, discuss, and exchange ideas. In 2012 she was the chair of the 48<sup>th</sup> International Conference on Microelectronics, Devices and Materials with the Workshop on Ceramic Microsystems and she managed to attract many participants from Slovenian industry.

The world of science lost not only a great scientist, but also a woman who was able to accept challenges and solve them with optimism and a positive approach. One of her acquaintances from abroad wrote that she was a sunny personality. Professor Marija Kosec was, if I may borrow the words of the current President of the MIDEM Society, Professor Marko Topič, a bright star in the sky of Slovenian science.

She will be missed by her colleagues and friends both at home and in the international communities.

Prof. Dr. Barbara Malič Jožef Stefan Institute



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## MIDEM 2013

### 49<sup>th</sup> INTERNATIONAL CONFERENCE ON MICROELECTRONICS, DEVICES AND MATERIALS WITH THE WORKSHOP ON DIGITAL ELECTRONIC SYSTEMS

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