

# 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 Wrocławu.

**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 microengineering 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 microsystem applications: analytical chemistry, medical diagnosis, DNA sequencing, cell separation, microbiological analysis, high-throughput synthesis, environmental monitoring and others [3-6].

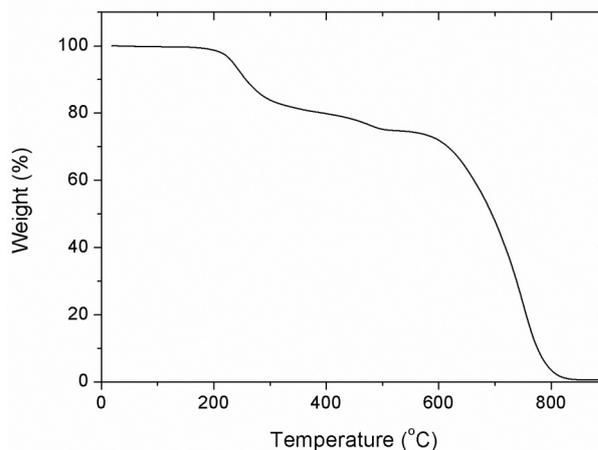
Nowadays fluidic microsystems are fabricated using silicon/glass [7], polymer [8], PCB (Printed Circuit Board) [9] and LTCC (Low Temperature Co-fired Ceramics) [10-12] technologies. The main advantages of the LTCC in comparison to silicon, polymers and PCB are chemical inactivity, chemical resistance 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 three-dimensional (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 co-fired 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.



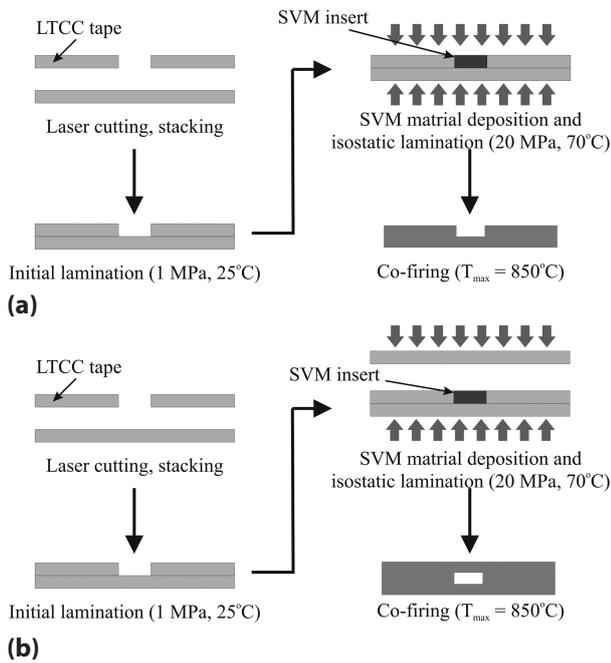
**Figure 1:** TG analysis results of the graphite-based SVM paste.

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.

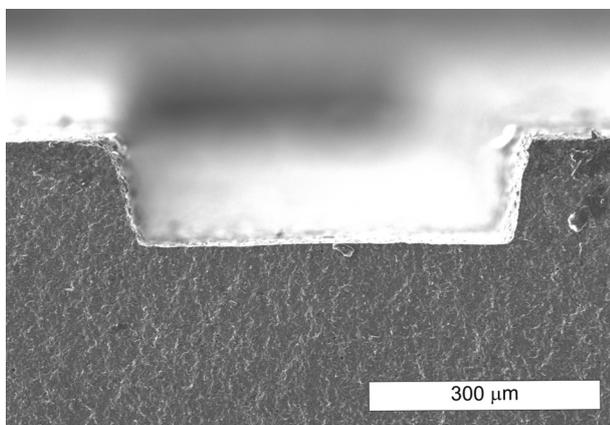
### 2.1. Define and fill

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.



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

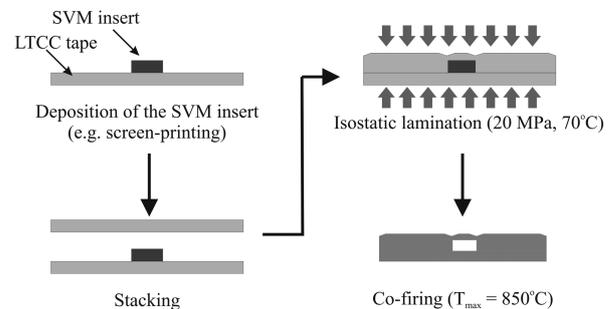
Moreover, the SVM paste shrinks after drying and carrier film ensures proper thickness of dried SVM. The SVM deposition process is called “screeding” in the literature [25]. In the next step the fluidic structure is sealed with a top LTCC tape layer. Second lamination is performed at pressure of 10-20 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.



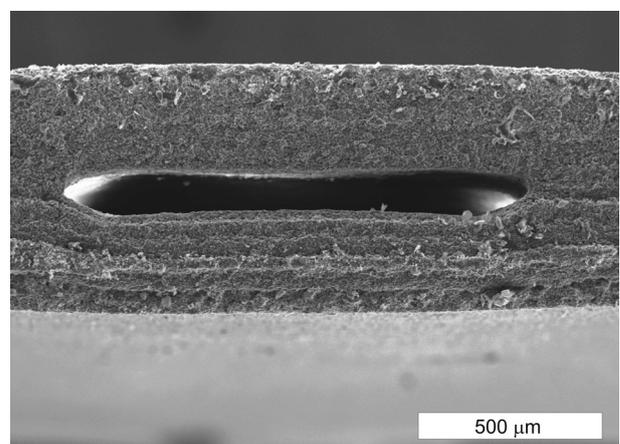
**Figure 3:** SEM image of the open channel made in the LTCC module using “define and fill” technique.

## 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.



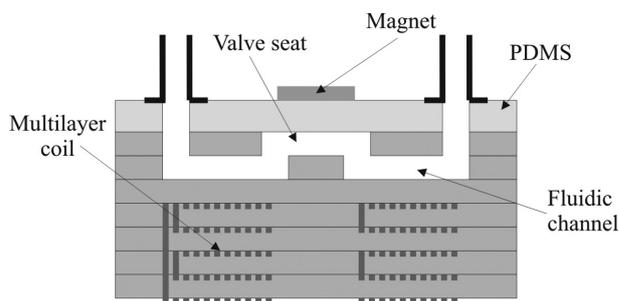
**Figure 5:** SEM image of the open channel made in the LTCC module using “collate and laminate” technique.

### 3. LTCC-based microfluidic systems

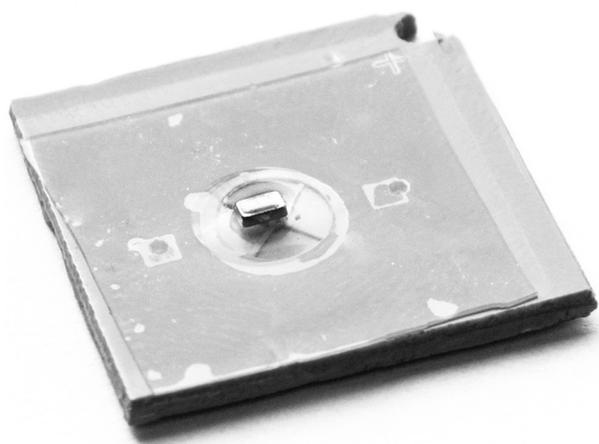
Modern microfluidic system is build of several functional blocks. These sub-systems are responsible for various functions: sample transport (micropumps and microvalves), preliminary preparation of the sample (micromixers), carrying out appropriate (bio)chemical reaction, product separation and detection. 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  $\mu\text{m}$  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  $\mu\text{m}$  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):

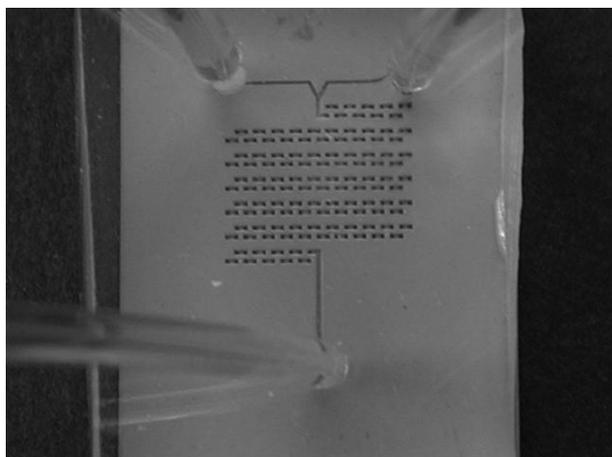
$$t_{diff} \propto \frac{D_h^2}{D} \quad (1)$$

where  $D_h$  is a characteristic dimension of the mixer (m) and  $D$  is a coefficient of molecular diffusion ( $\text{m}^2/\text{s}$ ). 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):

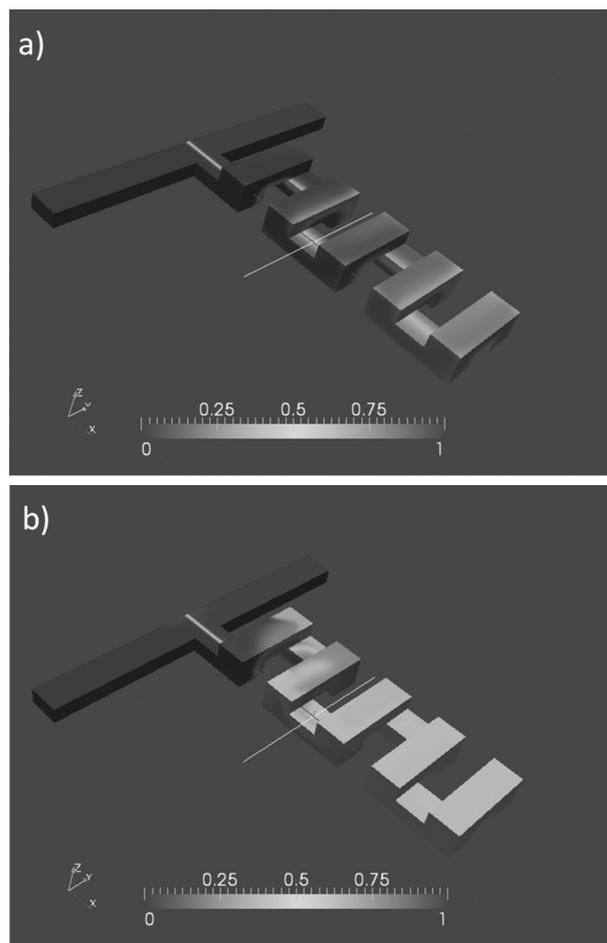
$$J = -D\nabla c \quad (2)$$

where  $c$  is particles concentration ( $\text{m}^{-3}$ ). 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 area 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 micro-channel. The Lorentz 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 Lorentz 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 Electronics 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.

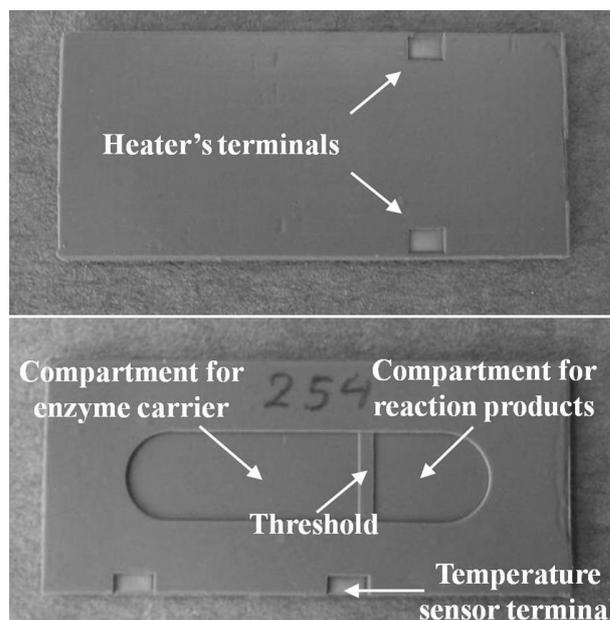
### 3.3. Microreactors

Microreactor technology has become very promising in the fields of chemistry, biotechnology and process engineering. 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 stand-alone 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.

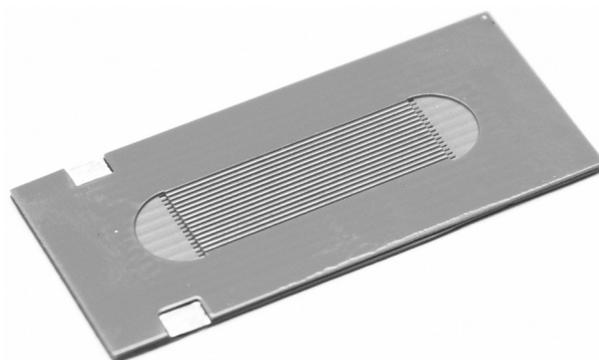
The presented microreactors were used for urea determination in biological fluids [34]. The principle of their operation is based on hydrolysis of urea catalyzed by urease. One of the reaction products are hydroxyl ions which are used for indirect determination of urea in the sample.

### 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.

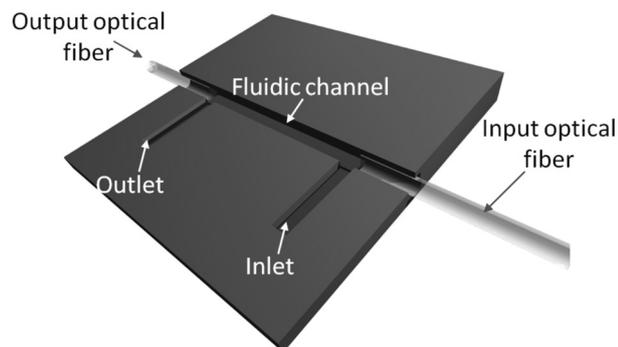


**Figure 10:** Batch type microreactor made with LTCC technology.

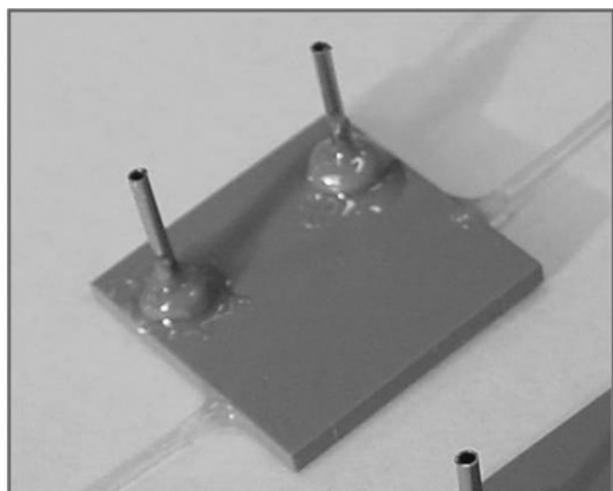


**Figure 11:** Flow-through microreactor made with LTCC technology.

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-to-voltage converter. Principle of operation is based on selective light absorption by the analyte. A green light ( $\lambda_{\text{max}} = 565 \text{ nm}$ ) 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:



(a)



(b)

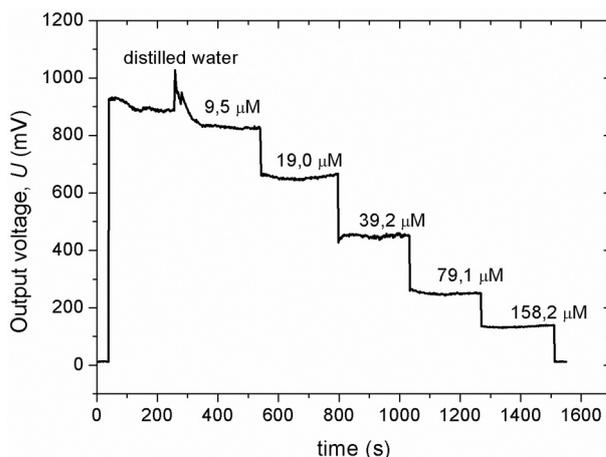
**Figure 12:** LTCC-based detection unit for absorbance measurements: (a) schematic view and (b) photograph.

$$A \equiv \log \frac{I_0}{I} = \epsilon c l \quad (3)$$

where  $I_0$  and  $I$  are intensities of the light before and after absorption,  $c$  is the molar concentration of the analyte (M),  $\epsilon$  is the molar absorptivity ( $\text{cm}^{-1} \text{M}^{-1}$ ) and  $l$  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:

$$A \equiv \log \frac{I_0}{I(c)} = \log \frac{U_0}{U(c)} \quad (4)$$

where  $U_0$  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 ( $\text{KMnO}_4$ ) 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  $\text{KMnO}_4$  test solution ( $\lambda_{\text{max}} = 565 \text{ nm}$ )

#### 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.

#### Acknowledgments

The financial support of the European Union through the MULTILAYER project (FP7-NMP4-2007-214122) is gratefully acknowledged.

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Arrived: 10. 08. 2012

Accepted: 25. 10. 2012.