# LTCC system for light absorbance measurement

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**Abstract**

*The paper describes technology of the LTCC structure which enables light absorbance measurements of liquid sample. The manufactured ceramic structure contains buried microfluidic channels. The structure consists of two co-fired glass windows which separate the light source and detector from the test solution. A construction of an electronic measurement system is described as well. The signal from three LEDs (Light Emitting Diode) – red, green and blue – can be used in the absorbance measurements. The light intensity is measured by the TCS3414CS (TAOS) color detector. Optical properties of the fabricated microfluidic LTCC system is investigated with several concentrations of potassium permanganate ($KMnO_4$) in water solution. The system can be applied in microbiology for constant monitoring of bacteria growth.*

Key words: microfluidic, light absorbance, LTCC

## 1. Introduction

There is a great interest in analytical microfluidic devices. These fast and reliable systems can find wide range of applications (e.g. environment monitoring). Miniature chips, which are able to perform at least one laboratory function, are known as Micro Total Analysis Systems (µTAS) or Lab-on-Chip (LOC) devices. There are many materials which can be applied in the realization of the LOC devices. Silicon and glass techniques are the most popular in this field. However, other materials like PDMS (poly(dimethylsiloxane)) or glass-reinforced epoxy laminate (e.g. FR4) are also used for the fabrication of the structures.

The LTCC (Low Temperature Co-fired Ceramics) technology can be applied for construction of miniature analytical system. The LTCC electronic structure is composed of dielectric tapes on which different conductive, resistive or dielectric films are deposited forming an electric circuit [1]. Then, the active electronic components are mounted on the surface [2]. However, the LTCC chip also can contain buried microfluidic channels and sensors. A wide range of analyses can be realized in such ceramic structure. One of them is light absorbance analysis.

The LTCC is an opaque material. In this case it is significant disadvantage because additional optical ports have to be manufactured. The simplest method of light coupling with opaque microfluidic structures is gluing of optical fibers in a post firing process [3]. However, this technique is problematic and it requires additional materials and technological steps.

The integration of transparent glass parts with LTCC structure is a promising method [4, 5]. It is possible to put glass part into green structure and simply co-fired the element in order to obtain LTCC glass solid structure. The main issue in this case is to use proper glass material. Coefficients of Thermal Expansion (CTE) of both materials (glass and ceramics) should be similar. The key to the successful glass window manufacturing in the LTCC structure is to use glass with proper softening point. On the one hand, the point needs to be lower than the LTCC shrinkage temperature in order to avoid deformation during sintering. On the other hand, the softening point needs to be as high as possible to achieve the minimal necessary viscosity of a liquid glass [4].

Light absorbance measurements are commonly used in microbiology for bacteria growth monitoring. Tests are usually made in Petri dishes. Presented fluidic system can provide a tool for constant solution monitoring.

## 2. Fabrication

The LTCC technology was used for fabrication of a microfluidic chip for the light absorbance measurements of liquid sample. The schematic view of the system is presented in Figure 1. It consists of inlet and outlet channel, one centimeter long absorption region, chamber for three different light sources and chamber for a light sensor. Moreover, it incorporates openings for glass plates which separate the light source and the detector from the test solution in the absorption region.

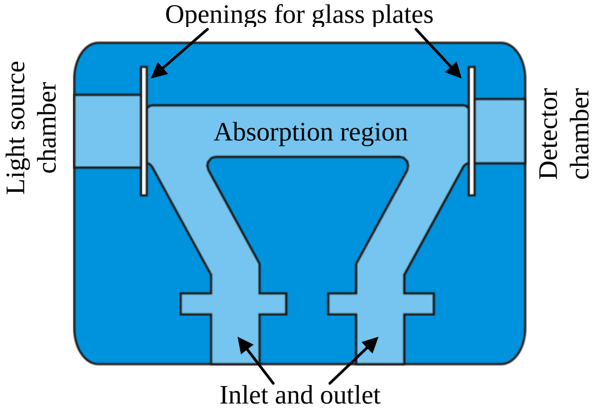
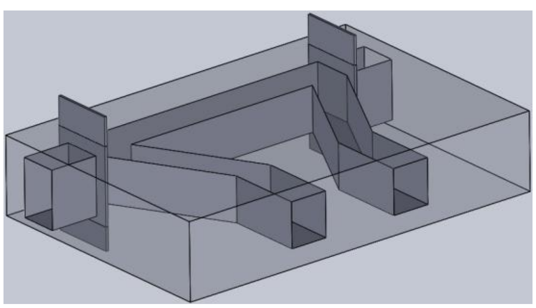



**Figure 1. Schematic view of the microfluidic system.**

The LTCC microfluidic chip was made of thirteen layers of DuPont™ 951 LTCC tapes. Each LTCC tape was 254 μm thick before firing. The Nd-YAG (Aurel NAVS 30) laser system was used for green tape layers patterning. Registration holes, channels, chambers and openings for glass plates were manufactured. Afterwards the ceramic layers were stacked together and laminated in an isostatic press. The lamination process was carried out at temperature of 70 °C and pressure of 4 MPa. Then the glass plates were assembled into the LTCC structure and co-fired in a box furnace at a recommended two-step firing profile with a peak temperature at 850 °C.



A

B

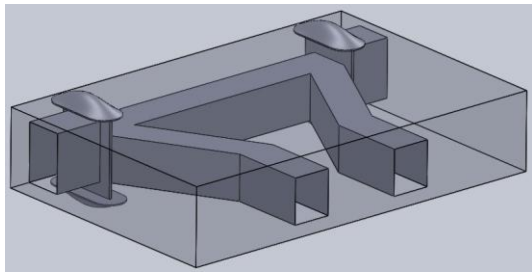


**Figure 2. Schema of glass plates set in LTCC structure; A – before firing, B – after firing (not in scale).**

The technology similar to the technique described in the paper [4] was used for manufacturing optical ports in the LTCC structure. The thin glass plates (160 μm) were inserted into the ceramic structure (Figure 2A). The glass plates closed the fluidic channel. During firing process the glass get soft and wet the ceramics. As a result glass and ceramics were combined (Figure 2B).

The LTCC structure for the light absorbance measurement is presented in Figure 3.

**Figure 3. The LTCC structure for the light absorbance measurement.**

The hermetic channel with glass windows was achieved. The great advantage of the construction was that the light source and light sensor did not have physical contact with the measured sample (Figure 4). Electronic components (light source, detector) were attached to the light ports as the external parts. The construction enabled easy replacement of fluidic chips. Thus, LTCC-glass fluidic structure could be disposable.

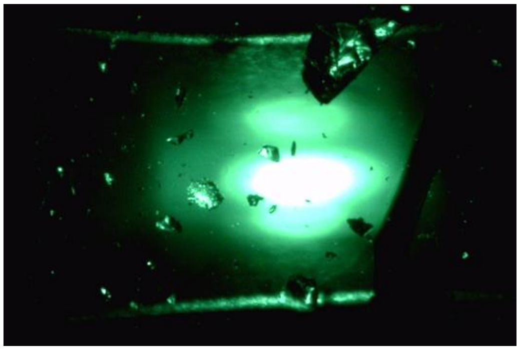
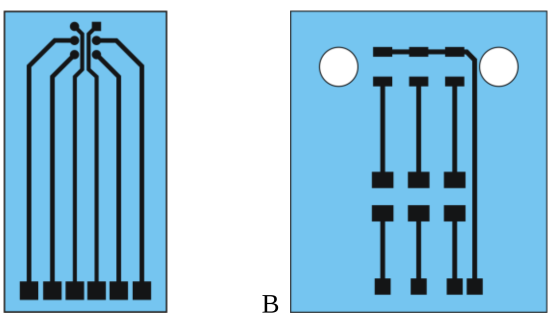


**Figure 4. View of the absorbance region in the LTCC structure through the glass window.**

Ceramic substrates for three different light sources and light detector were designed and manufactured (Figure 5). Four layers of DP 951 LTCC tape (254 μm thick) were used. The PdAg (DP 6146) thick film paste was screen printed through a 325 mesh stainless steel screen forming conductive lines at the top layer of the manufactured substrates. Additional 50 μm thick DP 951 LTCC tape was used as a protective layer for deposited conductive lines. The lamination process was performed at pressure of 20 MPa for 10 minutes. The co-firing process was performed as described above. Light from light emitting diodes (LEDs) can be coupled by light guide switch into one optical fiber and transferred to the optical port in LTCC microfluidic chip.



A B

**Figure 5. Design of light detector (A) and light sources (B) substrates (not in scale).**

Light sources and passive electronic components were soldered to the substrate using SMT (Surface-Mount Technology) method. In order to minimize the size of the structure 0603 and 0805 SMD components were chosen. The TCS3414CS component was mounted on the LTCC substrate using flip chip method with a dedicated solder reflow profile (Table 1). The picture of the part is presented in Figure 6.

**Table 1. TCS3414CS solder reflow profile [6]**

| | |
|---|---|
| Average temperature gradient in preheating | 2,5 °C/s |
| Soak time | 2 to 3 min |
| Time above 217 °C | max 60 s |
| Time above 230 °C | max 50 s |
| Time above $T_{peak}$ – 10 °C | max 10 s |
| Peak temperature $T_{peak}$ | 260 °C (–0 °C/+5 °C) |
| Temperature gradient in cooling | max 5 °C/s |

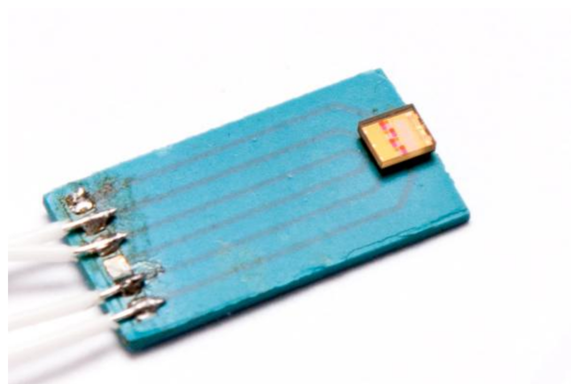


**Figure 6. Surface mounted TCS3414CS color detector.**

An X-ray Computed Tomography was used for examination of proper position and solder quality of the light detector. The X-ray image of mounted electronic component is presented in Figure 7. The TCS3414CS was well positioned and soldered to the substrate. The element was connected to ATmega microcontroler and short-circuits were not observed.

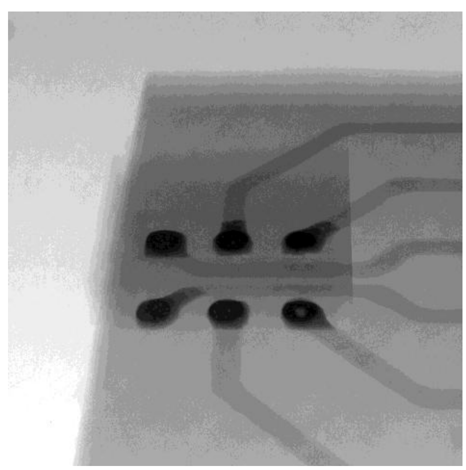


**Figure 7. X-ray picture of assembled color detector.**

## 3. Measurement system

Three different light emitting diodes (OF-SMD 1608R/G/B, Optoflash) were used as a light source. The red diode was a GaAsP/GaP device with a peak wavelength at $\lambda_p$ = 624 nm and a spectral halfwidth equal to $\Delta\lambda½$ = 35 nm. GaP was applied in the green LED. It had a peak wavelength at $\lambda_p$ = 515 nm and spectral halfwidth $\Delta\lambda½$ = 30 nm. The blue diode was made of GaN with a peak wavelength at $\lambda_p$ = 624 nm and spectral halfwidth equal to $\Delta\lambda½$ = 30 nm. All LEDs were mounted on the ceramic substrate described above.

The TCS3414CS (TAOS, USA) chip was used as a light detector. It is a high tech device which is quite new product on the market. It is a sophisticated device enclosed within small package (2.1 × 1.9 × 0.8 mm). It is designed to accurately derive the color chromaticity and luminance of ambient light. The device includes an 8 × 2 array of filtered photodiodes, analog-to-digital converters, and control functions on a single monolithic CMOS integrated circuit. The 12 photodiodes are covered by optical filters – 4 have blue filters (peak wavelength $\lambda_p$ = 470 nm, spectral halfwidth $\Delta\lambda½$ = 55 nm), 4 have green filters (peak wavelength $\lambda_p$ = 524 nm, spectral halfwidth $\Delta\lambda½$ = 45 nm), 4 have red filters (peak wavelength $\lambda_p$ = 640 nm, spectral halfwidth $\Delta\lambda½$ = 35 nm). The device sends 16 bits value of light to digital conversion. Communication with device is realized by Two-Wire Interface (TWI) protocol.

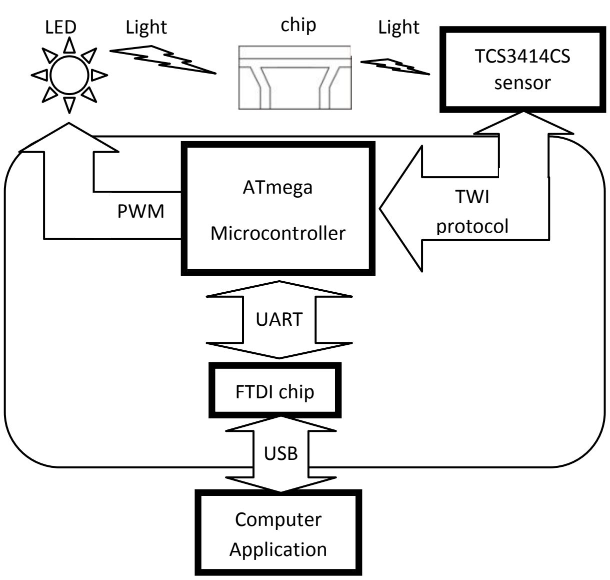



**Figure 8. Schema of the measurement system.**

Digital electronic circuit was constructed. The electronic system enabled communication between TAOS device and computer application. The user could easily set conversion parameters and luminance of light source with the computer program. The schema of the measurement system is presented in Figure 8.

## 4. Measurements

Absorbance measurements of several different potassium permanganate ($KMnO_4$) concentrations were performed to examine properties of the manufactured LTCC microfluidic device. Due to partially or completely opaque properties of most biological cells light absorbance measurements are used in microbiology e.g. for bacteria growth monitoring [7].

Distilled water was used in preparation of the test solutions. A 380 µM concentration of potassium permanganate solution was prepared which served as a base solution. Five concentrations in range from 60 to 300 µM were prepared and tested. The maximum solubility of $KMnO_4$ in water equals to 6,38 g/100 mL at temperature of 20 °C. The maximum light absorption of $KMnO_4$ occurs for wavelength $\lambda_{max}$ = 535 nm.

Test solutions were pumped to the ceramic chip with the Perfusor® syringe pump in order to examine properties of the fabricated LTCC microfluidic chip. Measurements of each solution absorbance were made in approximate five minutes intervals. The microfluidic chip was purged with distilled water between measurements. Test measurements were performed with the green LED ($\lambda$ = 515 nm) light source because the maximum light absorption of test solutions. Incident light was partially absorbed in the absorption region. Then the intensity of the non-absorbed light was measured with TCS3414CS. The response time of the LTCC microfluidic system is shown in Figure 9.

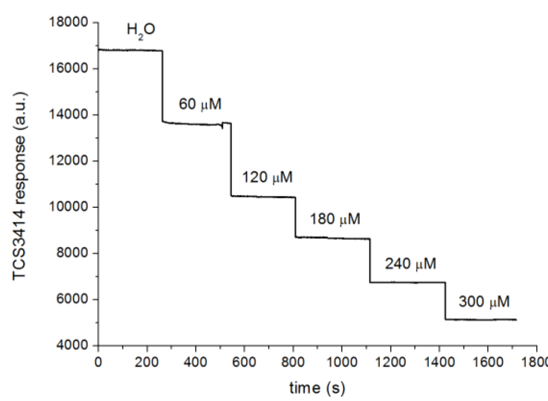



**Figure 9. The LTCC microfluidic system response time.**

The measured light intensity was proportional to the concentrations of the test solutions. Moreover, high measurements stability was observed.

The repeatability of the measurements was examined in order to determine the reliability of the ceramic microfluidic system. Water and 180 μM solution of $KMnO_4$ were used during the test. Both liquids were used alternately. The experiment has shown very good repeatability of the measurements (Figure 10). The TCS3414CS response indicates good stability during real-time measurements.

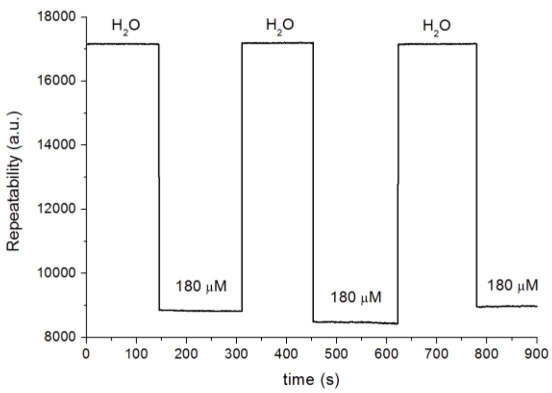



**Figure 10. Repeatability of measurements.**

According to the Beer-Lambert's law absorbance magnitude can be presented as logarithm of the light source and non-absorbed light intensities ratio. Absorbance magnitude of the $KMnO_4$ solution concentration was determined with following equation:

$$A = \log\left(\frac{I_0}{I}\right) \quad (1),$$

where $I_0$ is an output signal for distilled water and $I$ is an output signal for the test solution concentration.

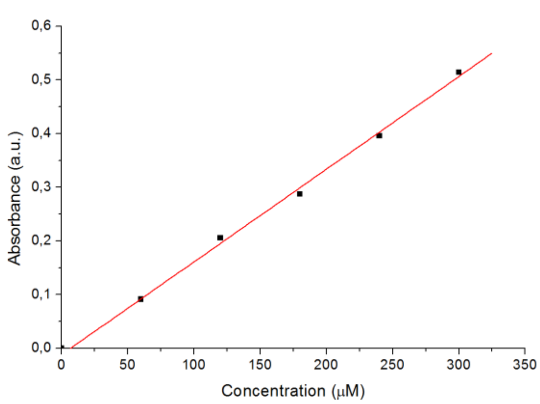



**Figure 11. Calibration curve.**

The calibration curve of the LTCC microfluidic system is presented in Figure 11. It is possible to evaluate solution concentration according to its light absorbance.

## 5. Conclusions

The ceramic microfluidic system for light absorbance measurement was designed and manufactured. Moreover, integration of the glass plates with the LTCC structure was presented.

The performance of the fabricated ceramic system was investigated with several different solution concentrations of $KMnO_4$ in distilled water. Measured light absorbance was proportional to the concentration of potassium permanganate solutions.

The reliability of the LTCC system was examined. The experiment has shown very good repeatability of the measurements. The TCS3414CS response indicated good stability during real-time measurements.

The manufactured ceramic system consists of three different light sources and can be used as a universal system for light absorbance measurements. The fabricated LTCC microfluidic chip can be assigned for bacteria growth monitoring.

## Acknowledgment

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