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LTCC Microfluidic System

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A simple and inexpensive low-temperature cofired ceramic (LTCC) microfluidic device with integrated optical fibers is designed, manufactured, and tested with positive results. Fluidic channels, mixer, detector, optical fiber, light source, light detector, heater, and temperature sensor are integrated in one LTCC module. The optical system in the LTCC microsystem permits measurements of light transmittance and fluorescence. The design, technology, and results of the module's evaluation are presented.

Introduction

This paper presents a microfluidic system that uses low-temperature cofired ceramic (LTCC) technology. This technology has been used in microelectronics since the end of the 1980s.^{1,2} The LTCC technology is well established both for low-volume, high-performance (military, space) and high-volume, low-cost (portable wireless, automotive) applications. The market for LTCC devices is growing very fast due to very good electrical and mechanical properties, high reliability, and stability as well as the possibility of making threedimensional (3D) microstructures. Special methods of micromachining are used for preparing 3D structures, channels, and cavities inside the LTCC modules.^{2–8} New LTCC modules are becoming more and more sophisticated. They contain electronics, cooling or heating systems, sensors and actuators, and microsystems.^{1,2,9–13} Microfluidic applications of LTCC devices are particularly interesting.^{14–18} Sophisticated micro-electromechanical systems and micro-opto electromechanical systems packages made using LTCC technology integrate electronic measurement, control, and signal conditioning circuits. Moreover, electrical, optical, gas, and fluidic network systems are realized in one package.¹⁹

An LTCC microfluidic system is presented in this paper. Similar systems made of silicon and glass²⁰ or poly(dimethylsiloxane)²¹ materials have been described recently. The advantages of the LTCC structure, in comparison with silicon, are a much lower price and a shorter development time. Moreover, the fabrication process of LTCC microsystems is much simpler. The possibility of fluidic channels, heater, sensors, electronics, and package integration in one LTCC module is the main advantage of this technology over silicon, glass, and polymer technologies. Thick film materials offer the possibility of manufacturing not only a network of conductive paths in a package but also of other electronic components, sensors, and microsystems.

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An LTCC microfluidic device with a fluid mixing meander, a Y-shaped reagent junction, a channel for optical detection, optical fibers, fluid input/output, a heater, a temperature sensor, a and dedicated temperature controller was designed, manufactured, and tested. The configuration of connected optical fibers allows measurements of both light transmittance and fluorescence intensity. The device was used for chemical analysis of liquids. The microfluidic system was connected to typical analytical UV-Vis and spectrofluorimetric microanalytical systems via long optical fibers. Light transmittance and fluorescence measured in the system were presented in Golonka et al.¹⁸ A similar system containing short quartz optical fibers, together with a light source and detector integrated with the LTCC module, is presented in this paper. Microfluidic system technology, quartz optical fiber integration methods, and temperature controller are described. In order to verify the measurement efficiency of light transmittance, solutions of Ponceau IV R were pumped into the LTCC microsystem using a peristaltic pump. The optical detection was performed at $\lambda = 502 \text{ nm}$ using optical fibers. A high-efficiency LED was applied as a light source and this transmitted light via one optical fiber to the detection channel. The opposite fiber was connected to the integrated light detector.

Microfluidic System

The LTCC microfluidic system contained a fluid channel inlet and outlet, a mixing meander, a detection sector with integrated optical fibers for light transmittance and fluorescence measurements, a heater, and a temperature sensor (Fig. 1). Two standard multimode 62.5/125 optical quartz fibers were positioned inside the detection microchannel. A high-efficiency LED was applied as a light source in the system described in this paper. This transmitted light via one optical fiber to the detection channel. The opposite fiber was connected to the light detector (Fig. 2).

Fluidic System Technology

Six layers of DuPont 951 A2 foil were used to make the LTCC modules. The thickness of each foil was equal to 137 μ m after firing. A thick film heater was made by screen printing using PdAg DuPont 6146 ink on the bottom face of the module, and a platinum resistance

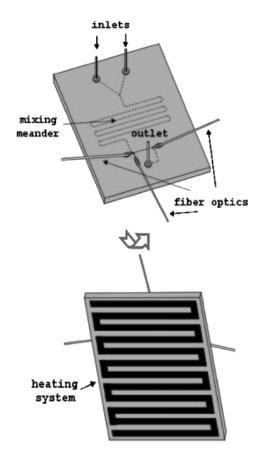


Fig. 1. Low-temperature cofired ceramic microfluidic system (not to scale).

temperature sensor was also located here. The temperature sensor and the heater were connected to a temperature regulator in order to keep the temperature in the microreactor constant. The vias, fluid channels, and channels for optical fibers were made inside the green LTCC foils using an Nd-YAG laser (Aurel NAVS 30 laser trimming and cutting system, AUREL, Modigliana, Italy). Moreover, the laser system was used for patterning the heater. The special construction of the optical fiber channels allowed precise positioning of the fibers. In the subsequent step, the LTCC foils were stacked into one module in the proper order, pressed in an isostatic press, and then cofired in air at a typical temperature profile (maximum temperature 875°C). Two standard multimode 62.5/125 quartz optical fibers were positioned with a high precision to ensure a 1 cm optical path length and then glued to the cofired structure. The inlet and outlet of the fluid channels were also glued to the cofired structure.

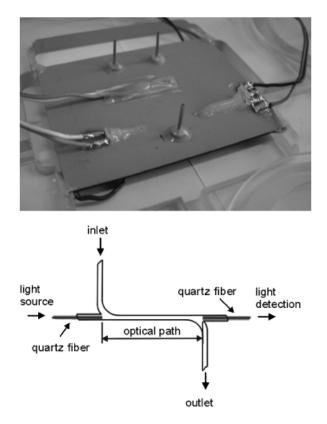


Fig. 2. Microfluidic system with an integrated light source (LED) and a light detector (not to scale).

Control of the fluid temperature within the microanalytical system was very important for its proper working. The integration of the heater and temperature sensor elements inside the LTCC module gave the desired temperature level and distribution. The temperature sensor and the heater were connected to a microprocessor-based temperature regulator in order to maintain a constant temperature in the microreactor.

Optical Fibers Integration

Before producing the LTCC microfluidic module, the problem of optical fiber integration had to be solved. At first, we tried to cofire the quartz fibers together with the LTCC structure. A glass thick film was used as an adhesive.

Test structures with both surface and buried optical fibers were examined.

Surface fiber preparation: optical fibers were mounted using a glass film on a cofired LTCC surface, and

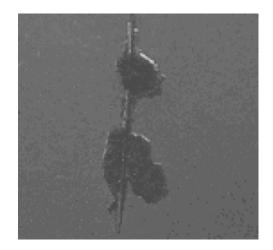


Fig. 3. Picture of a surface quartz optical fiber positioned on a glass film made on a fired low-temperature cofired ceramic structure.

then a firing process was carried out at a typical thick film firing profile (Fig. 3).

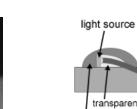
The buried fiber preparation was as follows:

(a) fibers were positioned in the LTCC module and then laminated and cofired with the whole structure (Fig. 4—delamination problem occurred),

(b) fibers were positioned in the channel inside the LTCC module, laminated together, and cofired (Fig. 5—fiber position not stable), and



Fig. 4. Optical microscope picture of a cofired quartz optical fiber made inside a low-temperature cofired ceramic structure (crosssection), delamination problem visible.



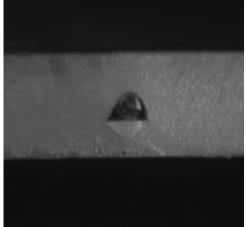


Fig. 5. Optical microscope picture of a cofired quartz optical fiber positioned in the channel made inside a low-temperature cofired ceramic structure (cross-section).

(c) fibers were positioned on a glass film inside the channel within the LTCC module and laminated and cofired together (Fig 6).

The results of our investigation were not satisfactory. The quartz optical fibers running outside the LTCC material were too weak. They broke very easily after the cofiring process.

On the basis of these experiments, we decided to integrate the optical quartz fibers within the fired LTCC

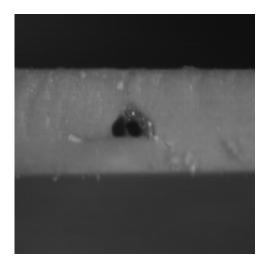


Fig. 6. Optical microscope picture of a cofired quartz optical fiber positioned using a glass layer in the channel made in a low-temperature cofired ceramic structure (cross-section).

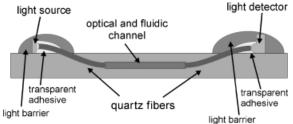


Fig. 7. Schematic drawing of an optical system integration (not to scale).

structure. The fibers were installed inside specially made microchannels (Fig. 7). The microchannel design ensured that the optical fibers were positioned with high precision and ensured proper integration of the fibers with the light source and detector on the LTCC surface. The fibers were glued to the cofired structure (Fig. 8).

Temperature Controller

As temperature level and distribution were important for proper functioning of the fluidic microsystem, a dedicated temperature controller was designed. It was based on the well-known ATmega 8L microcomputer (ATMEL, San Jose, CA) with AVR architecture. The controller is illustrated in Fig. 9.

The temperature control loop consisted of the following modules: a microprocessor-based controller, an R/I converter (4–20 mA), a Pt100 temperature sensor, a specially designed integrated heater, and an FET transistor (actuator). Temperature measurement and temperature control were embedded in a microcontroller algorithm. A pulse width modulation signal was used to drive an FET transistor and control the power delivered to the heater.

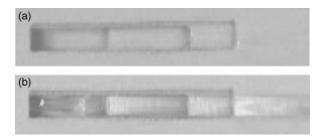


Fig. 8. Optical microscope picture of a microchannel for optical fibers positioning: (a) empty, (b) with an optical quartz fiber.

In the present stage of the investigation, an entire controller was fabricated on a separate PCB substrate, but in the future, thanks to the flexibility of LTCC technology, the controller will be placed in the same module as the microfluidic LTCC structure.

The controller was able to measure temperature in the range of 25–80°C with a 10-bit resolution. The R/I converter based on XTR103 allowed very precise tem-

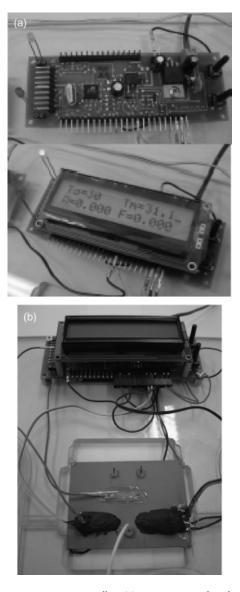


Fig. 9. Temperature controller: (a) microprocessor-based controller, (b) controller connected to the microfluidic low-temperature cofired ceramic module.

perature measurements with additional linearization of the Pt100 sensor.

As the microcontroller is very well equipped with sophisticated internal peripherals, it is possible to use it as an absorbance measurement unit. It is also possible to use it for communication with other control units (e.g., PC).

Optical Detection Circuit

The microsystem described was designed to detect light absorbance in a 10 mm optical path as a useful signal for measuring the concentration of a variety of substances in test solutions (Fig. 2). Typically, this kind of task is performed in laboratories using specially designed equipment. The main objective of our work was to establish a light transmittance measurement microsystem using low-cost optoelectronic components.

As a light source, SMD EL-12-21-UBGC LED (Everlight Electronics Co., Ltd., Taipei, Taiwan) was used. It is a high-performance electro-luminescence diode with a nominal wavelength equal to 502 nm.

The light detection circuit consisted of a high-sensitivity light to voltage converter TSLG 257 (TAOS Inc., Plano, TX). This converter combines a photodiode, optical filter, and transimpedance amplifier in a single monolithic CMOS integrated circuit. Its output voltage is proportional to the input light intensity.

Both the light source and the detector were attached to the input and output quartz fiber using a polymeric, transparent adhesive. It assured good optical coupling and mechanical stability of the optical detection circuit.

Experimental Procedure

The microfluidic system described was experimentally tested. It was applied for concentration measurement of food-coloring dye (Ponceau IV) in a water solution. It is known that the chosen dye has an absorbance maximum at a wavelength equal to 507 nm $(\pm 5 \text{ nm})$, which is sufficient for measurements at 502 nm (determined by the applied LED light source).

Because of the very small output signal level from the optical detector (caused by the small diameter of the quartz fibers used, and imperfect light coupling), special measurement conditions had to be assured. An AC actuation signal was used for supplying the LED in order

to 28 mA.

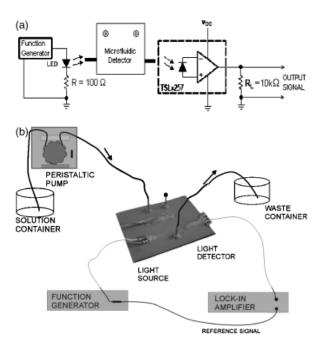


Fig. 10. Set-up: (a) schematic of the test circuit (b) schematic view (not in scale).

to eliminate the influence of external light source constant radiation. Because of the very small signal to noise ratio, lock-in amplification had to be used. In the experiments described, lock-in nanovolt meter Unipan type 232B (UNIPAN, Warsaw, Poland) was used. The LED was acted upon by a square-shaped signal with a 50% duty ratio and a frequency equal to 157.5 Hz. The same signal was applied for lock-in

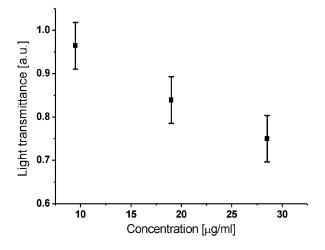


Fig. 11. Transmittance measurements of Ponceau IV solution.

reference. The maximal LED supply current was equal

Three concentrations, equal to 9.5, 19.0, and $28.5 \,\mu$ g/mL of Ponceau IV in distilled water, were examined. As the testing solutions were mixed in external equipment, one of the system inlets became clogged and the already prepared solution was pumped into the detection system using the remaining inlet (Fig. 10). In the experiments presented, a peristaltic pump was used (MasterFlex C/L, Cole Parmer Int., Vernon Hills, IL). It assured a constant fluid flow equal to 88 μ L/min. After the measurement of each concentration, the fluidic channels were purged using pure distilled water.

The results of light transmittance measurements versus Ponceau concentrations are given in Fig. 11.

Conclusion

A simple and inexpensive LTCC microfluidic device with integrated optical fibers was designed, manufactured, and tested with positive results. The optical system described, integrated with the LTCC microsystem, allowed measurements of light transmittance.

Various methods of quartz optical fiber integration were tested. Cofiring of fibers with the LTCC module gave no satisfactory results. Therefore, we decided to glue fibers to the cofired LTCC structure inside specially made microchannels. The design ensured that the optical fibers were positioned with a high precision inside the channel and that there was proper integration of the fibers with the light source and detector on the LTCC surface. Both the light source and the detector were attached to the input and output quartz fiber using a polymeric, transparent adhesive. It assured good optical coupling and mechanical stability of the optical detection circuit. As a light source, an SMD EL-12-21-UBGC LED was used. The light detection circuit consisted of a high-sensitivity light to voltage converter TSLG 257.

As temperature level and distribution were important for proper functioning of the fluidic microsystem, a special heater and a dedicated temperature controller were designed. The controller was based on an ATmega 8L microcomputer with AVR architecture, and was able to measure and control temperature in the range of 25– 80°C with a 10-bit resolution.

The microfluidic system described was tested experimentally. Light transmittance dependence upon the concentration of food-coloring dye (Ponceau IV) in water solution was measured. Because of the very small output signal level from the optical detector, lock-in amplification had to be used. An AC actuation signal was used for the LED power supply in order to eliminate the influence of constant radiation of an external light source. The measurements of the optical detectors in our LTCC module indicated that the light source introduced insufficient light into the optical fibers and the detecting device. It was possible to observe signal changes; however, the noise level was quite high. In order to solve the above-described drawbacks, optical fibers with a higher numerical aperture can be applied. The results of our experiments with plastic fibers (0.75 mm diameter) instead of 62.5/125 optical quartz fibers were presented in.²² The higher numerical aperture of optical fibers introduced more light into the detection channel.

The possibility of integrating fluidic channels, heaters, sensors, and electronics into one module is a great advantage of LTCC technology. Integration of fluidic channels, a heater, a temperature sensor, control and measurement electronics, and a light detection system (optical fibres, LED, photodetector) into one LTCC module simplifies the measurement setup significantly. Because microchannel geometry modification is easy, the LTCC micromodule can be adapted to different analytical methods. The fabrication process of LTCC microsystems, in comparison with a silicon one, is much simpler and cheaper.

The constructed device is found to be very useful in chemical analysis. Integration of optical fibers, a light source, and a light detector in one structure is very important in the case of on-line monitoring devices.

References

- L. J. Golonka, A. Dziedzic, J. Kita, and T. Zawada, "LTCC in Microsystems Application," *Informacije MIDEM 32/4* 272–279 (2002).
- L. J. Golonka, "Low Temperature Co-fired Ceramic (LTCC) Technology in Microelectronics," *Proceedings of the Symposium on Processing of Electroceramics*, Bled, Slovenia, 313–329, 2003.
- J. Kita, A. Dziedzic, L. J. Golonka, and T. Zawada, "Laser Treatment of LTCC for 3D Structures and Elements," *Microelectron. Int.*, 19 14–18 (2002).
- M. R. Gongora-Rubio, P. Espinoza-Vallejos, L. Sola-Laguna, and J. J. Santiago-Aviles, "Overview of Low Temperature Co-Fired Ceramics Tape Technology for Meso-System Technology (MsST)," *Sensors Actuat. A Phys.*, 89 222–241 (2001).
- 5. R. C. Colon, R. Furlan, E. W. Simoes, R. E. Bruzetti Leminski, M. R. Gongora-Rubio, and J. J. Santiago-Avilés, "Development of Micromolds

and Application with LTCC Substrates to Obtain Microfluidic Devices," *Proceedings of IMAPS Conference on Ceramic Interconnect Technology*, Denver, April, 139–143, 2004.

- H. Birol, T. Maeder, C. Jacq, and P. Ryser, "3-D Structuration of LTCC for Sensor Micro-Fluidic Applications," *Proceedings of European Microelectronics Packaging and Interconnection Symposium*, Prague, June, 366–371, 2004.
- M. Massiot and B. Roesner, "New 3D Possibilities of LTCC Technology," *Proceedings of 14th European Microelectronic and Packaging Conference*, Friedrichshafen, Germany, June, 25–28, 2003.
- J. Kita, A. Dziedzic, L. J. Golonka, and A. Bochenek, "Properties of Laser Cut LTCC Heaters," *Microelectron. Reliab.*, 40 1005–1010 (2000).
- H. Teterycz, J. Kita, R. Bauer, L. J. Golonka, B. W. Licznerski, K. Nitsch, and K. Wiśniewski, "New Design of an SnO2 Gas Sensor on Low Temperature Cofiring Ceramics," *Sensors Actuat. B Chem.*, 47 100–103 (1998).
- M. Gongora-Rubio, L. M. Solá-Laguna, P. J. Moffett, and J. J. Santiago-Avilés, "The Utilisation of Low Temperature Co-Fired Ceramic (LTCC-ML) Technology for Meso-Scale EMS, a Simple Thermistor Based Flow Sensor," *Sensors Actuat. A Phys.*, 73 215–221 (1999).
- D. L. Wilcox, "Multilayer Ceramic Microsystems Technology—An Overview," Proceedings of the 13th European Microelectronic and Packaging Conference, Strasbourg, May, 115–117, 2001.
- G. Wang, K. Saxena, F. Barlow, S. Ang, and A. Elshabini, "A Microjet Array Air Impingement Cooling Package Fabricated Using Low Temperature Co-Fired Ceramic," *Proceedings of the IMAPS Conference on Ceramic Interconnect Technology*, Denver, 77–82, 2004.
- J. J. Santiago-Avilés, M. R. Gongora-Rubio, P. Espinoza-Vallejos, and L. Sola-Laguna, "Sensors, Actuators and Other Non-Packaging Application of LTCC Tapes," *Proceedings of the IMAPS Conference on Ceramic Interconnect Technology*, Denver, 7–12, 2004.
- L. Rebenklau, K-J. Wolter, and S. Howitz, "Realization of Hybrid Microfluidic Systems Using Standard LTCC Process," *Proceedings of the 50th ECTC IEEE*, Las Vegas, 1696–1700, 2000.
- P. Wasilek, L. J. Golonka, A. Gorecka-Drzazga, H. Roguszczak, and T. Zawada, "LTCC Liquid Conductivity Detector," *Proceedings of the 26th International Spring Seminar on Electronics Technology*, High Tatras, Slovakia, May, 202–206, 2003.
- B. Smith, M. Pleskach, and C. Gamlen, "Microfluidics for Thermal Management: Applications, Fabrication and Demonstration in LTCC," *Proceedings of the IMAPS Conference on Ceramic Interconnect Technology*, Denver, 217–222, 2003.
- M. R. Gongora-Rubio, M. Bariatto Andrade Fontes, Z. Mendes da Rocha, E. Richter, and L. Angnes, "LTCC Manifold for Heavy Metal Detection System in Biomedical and Environmental Fluids," *Proceedings of the Eurosensors XVII*, Portugal, 823–826, 2003.
- L. J. Golonka, H. Roguszczak, T. Zawada, J. Radojewski, I. Grabowska, M. Chudy, A. Dybko, Z. Brzozka, and D. Stadnik, "LTCC Based Microfluidic System with Optical Detection," *Sensors Actuat. B*, B 11–112 396–402 (2005).
- 19. M. Schuenemann, K. A. Jam, V. Grosser, R. Leutenbauer, G. Bauer, W. Schafer, and H. Reichl, "MEMS Modular Packaging and Interfaces," *Proceedings of the 50th Electronic Components and Technology Conference IEEE*, Las Vegas, May, 681–688, 2000.
- S. Bargiel, A. Gorecka-Drzazga, J. Dziuban, P. Prokaryn, M. Chudy, A. Dybko, and Z. Brzozka, "Nanoliter Spectrofluorimetric Detector for Flow Systems," *Proceedings of the Eurosensors XVII*, Portugal, 669–672, 2003.
- M. Chudy, P. Prokaryn, A. Dybko, and Z. Brzozka, "Temperature Controlled Microfluidic PDMS Reactor," *Proceedings of the 7th International Conference on Miniaturized Chemical and Biochemical Analysis Systems*, Squaw Valley, 171–174, 2003.
- T. Zawada, K. Malecha, L. J. Golonka, J. Radojewski, H. Roguszczak, and M. Stefanow, "Integration of Light Detection in LTCC Fluidic Microsystems," *Proceedings of the 15th European Microelectronic and Packaging Conference*, Brugge, Belgium, 582–587, 2005.