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Integration of transparent glass window with LTCC technology for μTAS application

Pawel Bembnowicz*, Leszek J. Golonka

Faculty of Microsystem Electronics and Photonics, Wroclaw University of Technology, Wybrzeze Wyspianskiego 27, 50-370 Wroclaw, Poland

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Abstract

The LTCC substrate makes it possible to build various microsystems which integrate not only passive components such as resistors, capacitors and inductors but also 3D structures such as cavities and channels. Nevertheless non-transparency is a main limitation of the LTCC-based microfluidic systems. The goal of this paper is to present technology which allows an optical transparent element to integrate with LTCC co-firing process. A micrototal analysis system (μTAS), which is based on the LTCC–glass technology, enables optical measurements. The study shows that integration of sodium glass material is feasible not only with zero-shrinkage LTCC (HL 2000, HL 800) but also with a standard one (DP 951). A FEA (finite element analysis) is used to calculate stress inside the LTCC–glass structure. A series of LTCC–glass windows with different sizes and shapes is investigated to observe size limitation of the integration method. The example ceramic–glass structures (chambers, mixer) with glass windows are made in order to present the possibilities of this new technology.

Keywords: LTCC; Glass window; μTAS; Tension

1. Introduction

The miniaturization of total analysis systems (TASs) aims to solve some common drawbacks found in macroanalyzers. The advantages of μTAS are well known: low production costs, small sample volumes, low consumption of reagents and portability.[1] Most of the work in the literature is focused on microfluidics, as it is the basic platform needed for μTAS development. Silicon–glass chips have been the most widely used elements for this purpose, in part because glass is optical transparent and silicon bulk molding techniques are well known. Transparent walls of device make possible direct optical measurements of sample inside a chip. Moreover, microsystem, which is based on transparent substrate, enables simple addition to optical detection equipment.

Recently, LTCC microfluidics devices have shown a large number of advantages over those manufactured in glass or silicon, including the fast prototyping of complex 3D structures, the multilayer approach and lower cost of production. Moreover, a ceramic substrate is versatile, chemically robust and perfectly compatible with screen-printing techniques, allowing the integration of electronic circuits with surface mounted devices. Nevertheless ceramic material is non-transparent. This is a significant disadvantage from a μTAS application point of view. It is impossible to attach the structure directly to the optical detection systems. It is known few attempts to solve this problem. The scientists glued fibers[2], attached polymers[3], used optical glue[4] or used high pressure lamination to attach sapphire window.[5] However, all of the solutions have some undesirable features which cause problems with adapting it to the LTCC technology. Tan et al.[6] co-fire the zero shrink LTCC tape with a 0.5 mm thick borosilicate glass wafer in order to achieve the optimal co-firing profile for the transparent window and the ceramic chamber integration. Mülln et al.[7] compared properties of: fused silica, borosilicate and Pyrex (code 8511) glass with respect to LTCC–glass integration. However, unavoidable glass deformation during the process and a strong dependence on a temperature profile were disadvantages in all described experiments. Furthermore, all post-firing processes, which include a thermal treatment above glass softening temperature, intensify the deformation.

This paper presents research on novel integration technique of sodium glass with the LTCC multilayer module in order to

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* Corresponding author. Tel.: +48 071 355 4822; fax: +48 071 355 9718.
E-mail address: pawel.bembnowicz@pwr.wroc.pl (P. Bembnowicz).

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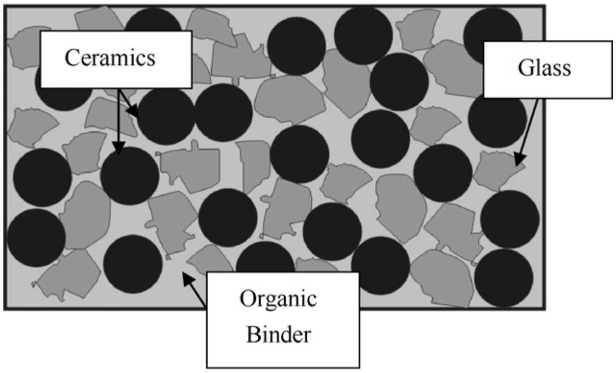
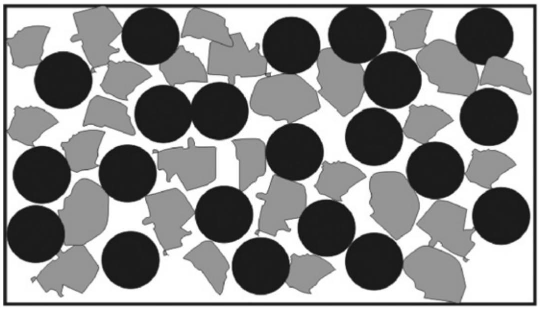
(a) In room temperature



(b) After organic binder evaporation



(c) Above glass binder softening temperature

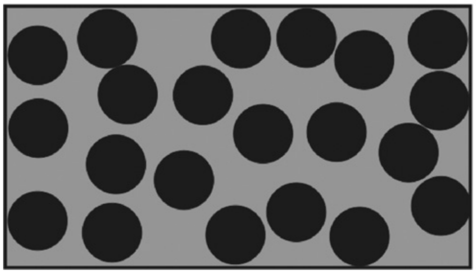


Fig. 1. Model of green tape composite materials during firing. (a) In room temperature, (b) after organic binder evaporation and (c) above glass binder softening temperature.

create a transparent window. This kind of structure can easily be connected to optical detection systems. LTCC and glass materials, combined and used in single technology, make it feasible to construct a leak proof, ceramic chamber with transparent walls. It opens a new application field for the LTCC structures as a base for optical analyses systems.

2. Idea of integration

The LTCC tapes are glass–ceramic composite materials. The ceramic filler is usually alumina (Al_2O_3) (45%). The composition also includes a glass binder (40%). A third component of the composite is an organic vehicle for binding and viscosity control of the tape before sintering (15%).[1,8] Fig. 1a demonstrates the model of green tape material before the firing process. The TGA (thermo gravimetric analyze) shows that the most significant weight lost during the firing process occurs at a temperature between 200 and 400 °C. At this temperature the organic binder is evaporated. Results of the TGA test for the DP 951 tape are demonstrated in Fig. 2. An additional derivative of the weight over temperature plot shows the temperature range where weight lost is the most significant.

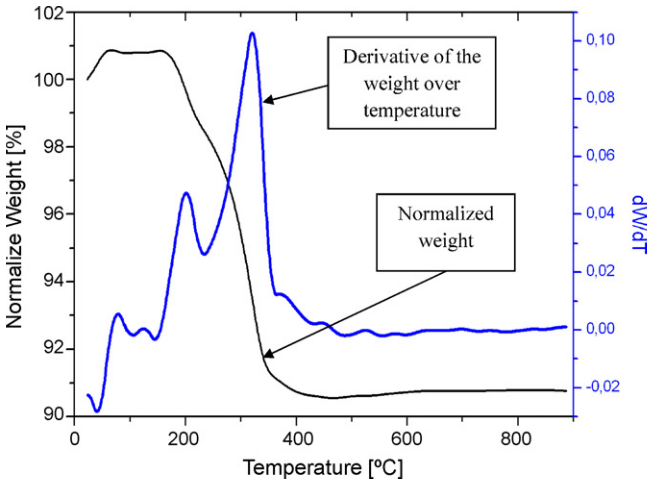


Fig. 2. The result of TGA test. Normalized weight lost (44.2 g = 100%) and derivative of the weight over temperature against temperature for DP 951P2 tape. TGA temperature rise 3 °C/min.

Then the structure consists of separated, solid particles of ceramics and glass (Fig. 1b). The glass material becomes liquid and tightly fills spaces between the ceramics particles as soon as firing temperature rises above the softening point (Fig. 1c). The structure shrinks as the filling process proceeds. According to Birol et al.[9] the DP 951 tape shrinkage begins at 670 °C and progresses proportionally till 880 °C.

The key to the successful glass wafer integration with an LTCC structure is a proper softening point of an additional glass wafer. On the one hand the point needs to be lower than the shrinkage temperature in order to avoid deformation in the LTCC structure 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.

The idea of the integration consists of using the thin glass wafer. The softening temperature point of glass is required to be range about 670–720 °C. The wafer needs to be located over the ceramic cavity (Fig. 3). As the firing temperature increases first the glass wafer becomes soft then the LTCC shrinkage starts. In this way the soft glass plate does not resist the shrinkage process. Simultaneously, the viscosity, in the maximum firing temperature, is high enough to create a surface tension (Fig. 4). The glass plate is thin. A mass of glass material is low. Thus the surface tension forces on the liquid glass membrane can be stronger than

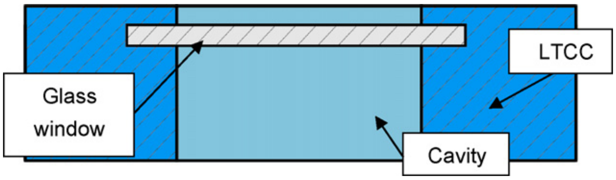


Fig. 3. Cross-section view of LTCC–glass structure.

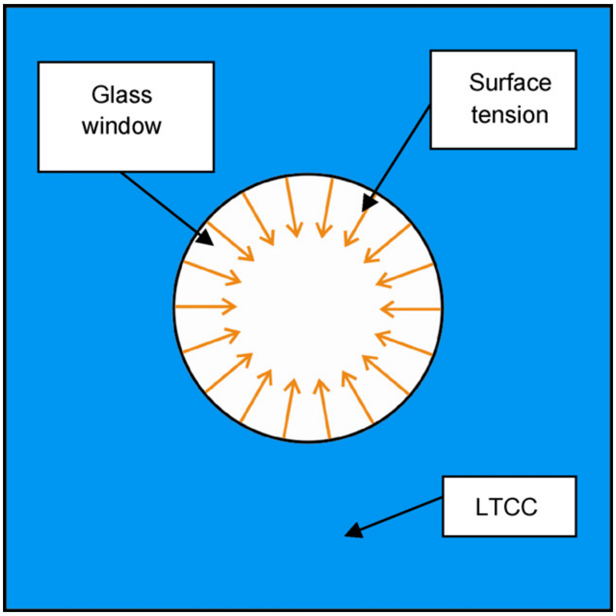


Fig. 4. Top view throw LTCC–glass structure with marked surface tension.

a force of gravity of liquid material. The surface tension forces are so strong that it can hold a soft glass membrane spread over the cavity. The thin glass membrane with an insignificant sag is created as a result.

3. Fabrication

The form was defined in an un-fired tape by Nd-YAG laser patterning (Aurel NAVS 30 laser trimming and cutting system). All the LTCC layers and glass wafer were aligned. The lamination stage is required before structures undergo the sintering process in a programmable box furnace. A classical lamination procedure, used to bind the layers, involves applying temperature (70 °C) and high pressure (200 bars) for time (10 min). In this case the high pressure lamination was inadmissible. The glass substrate was fragile at room temperature. Standard procedure would crush the glass wafer. Therefore one kind of cold and low-pressure lamination (CLPL) was used.[10] The cold chemical lamination (CCL) was applied.[11] A DuPont 4553 thinner acted as a solvent to a green tape sheet in the CCL process.

The zero-shrink (HL 2000 and HL 800) and standard (DP 951) LTCC tapes were tested. Cover glass slips were chosen. The light transmittance for the slips is 91.7%. An approximate chemical composition of the glass wafer is shown in Table 1. A softening point of the glass substrate was 720 °C. A thickness of the plate was about 150 μm.

Table 1
Glass wafer approximate chemical composition[12].

Silicon dioxide	SiO_2	72.20%
Sodium oxide	Na_2O	14.30%
Potassium oxide	K_2O	1.20%
Calcium oxide	CaO	6.40%
Magnesium oxide	MgO	4.30%
Aluminum oxide	AL_2O_3	1.20%
Ferric oxide	Fe_2O_3	0.03%
Sulfur trioxide	SO_3	0.30%

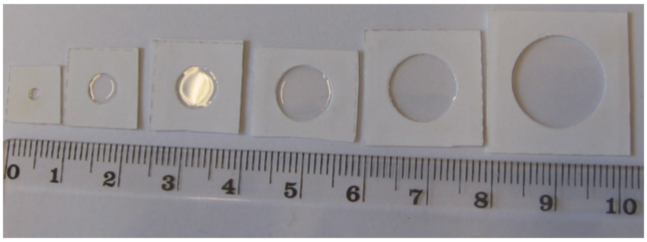


Fig. 5. An example of the round glass windows in ceramics (the scale in centimeters).

The size limitation of the sodium glass window integrated with different LTCC substrates (HL 2000, HL 800 and DP 951) was tested. A series of round and square glass windows were fabricated (Fig. 5). A side of a square and a diameter from 3 mm up to 21 mm with a 2 mm step were designed. Three different LTCC substrates were used. The firing profiles were different for all the LTCC tapes and were in line with the producers' data sheets. As a result the glass windows in LTCC substrate were successfully achieved.

An additionally post-firing test was made in order to check the thermal stability. The structures were heated up to 880 °C and cooled down to room temperature. The thermal treatment was repeated 10 times.

After firing processes a profile of glass surface was investigated. Fig. 6a demonstrates a surface scan of a 3 mm diameter glass window. A window cross-section profile is presented in Fig. 6b. Measurements were made by non-contact surface profiler (Taylor Hobson Talysurf CCI 3000).

Dependence between sag of glass plate and windows diameter is demonstrated in Fig. 7. A sag is understood as difference in position between the highest point and the lowest point on the top of glass plate. The sagging effect was measured by profiler.

4. Simulations

The sintering process includes the temperature treatment up to 900 °C. Therefore another important issue, which needs to be considered, is the TCE (thermal coefficient of expansion) differences of glass (Menzel's glass—9.1 ppm/°C) and ceramics (DP 951—5.8 ppm/°C, HL 2000—6.1 ppm/°C, HL 800—5.7 ppm/°C). The essential thermal range is between room and glass softening temperature. A FEA (finite element analysis) is used to calculate the stress inside the LTCC–glass model. The three-dimensional, 10 nodes, tetrahedral element with three degrees of freedom (translations in the nodal x, y, and z directions) is used. The tension is caused by the temperature change form 720 °C down to 20 °C and an incompatible TCE. TCE value for DP 951 is used in calculations. Fig. 8 shows a XY-shear stress on a quarter of the LTCC–glass chamber. The practical tensile strength of glass material is about 27–62 MPa. Thus the value of the maximum created tension is below the critical number.

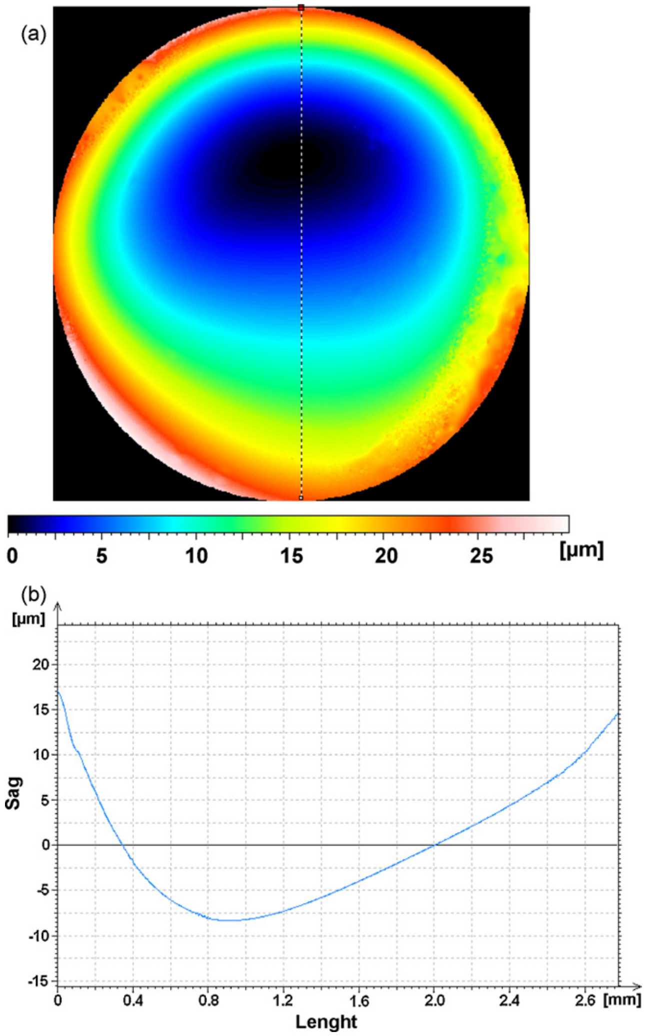


Fig. 6. Deformation of glass window. (a) Map of glass window surface and (b) profile of glass window surface.

5. Results

A series of LTCC–glass structures were successfully constructed. Both zero-shrink (HL 2000 and HL 800) and standard (DP 951) LTCC substrates proved to be suitable for this application. The measurements showed that size of window has influence to glass deformation. However, deformations of glass and LTCC were insignificant and acceptable. The maximum size of the glass window was not reached by the tested range. The shapes of the windows did not show significant influence on the maximum size reduction. Also the post-firing thermal test did not produce significant structural changes. The stress simulations showed that the tension created between two materials was lower than critical number.

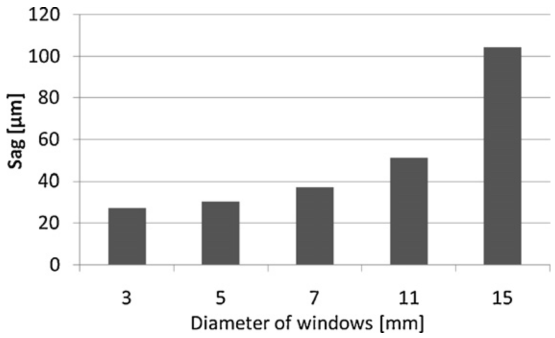


Fig. 7. Sagging effect vs. windows diameter.

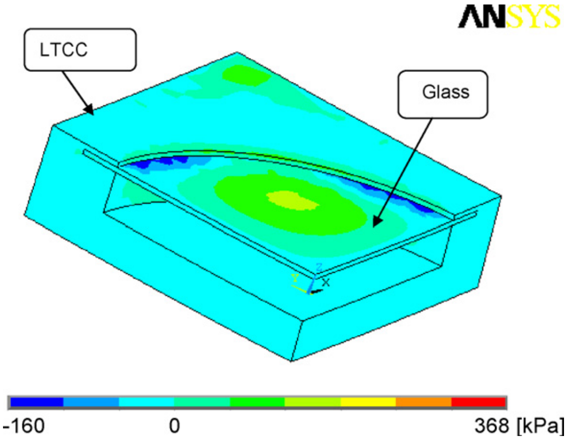


Fig. 8. The XY-shear stress on a quarter of the LTCC–glass chamber.

6. Application of the technique

The presented technology makes it feasible to build LTCC microfluidic chambers and channels with two transparent walls.

A transparent microfluidic chamber made of 10 DP 951 LTCC layers with two glass windows was produced. Fig. 9 presents the shape of the chamber's layers. The patterns were made in green LTCC tapes using a Nd-YAG laser (Aurel NAVS 30 laser trimming and cutting system). The glass wafers were situated between layers 2–3 and 9–10. All ceramic layers were laminated using CCL technique. Eventually the structure was co-fired at a standard for the DP 951 LTCC material temperature profile. Fig. 10 shows resulting test structure.

The same technology was applied to construct a Y-shape microfluidic mixer with two glass walled inlets and outlets. This construction enabled to observe a mixing process inside the ceramic structure. Fig. 11 shows the shape of all LTCC tapes which were used to manufacture Y-shape mixer. The result of mixer fabrication is presented in Fig. 12.

The previous examples of using the LTCC–glass technology demonstrate the application of the windows on horizontal walls. Whereas it is also possible to harness the technique in order to fabricate a vertical window in the LTCC structure. First eight LTCC green tape tapes were patterned by laser and laminated by CCL method. Fig. 13 shows the layers' shapes before lamination. Afterward the sodium glass wafer was located into a prepared section inside the green tape structure (Fig. 14). Finally LTCC–glass structure was sintered. Fig. 15 shows a real photo of a vertical window made in LTCC material.

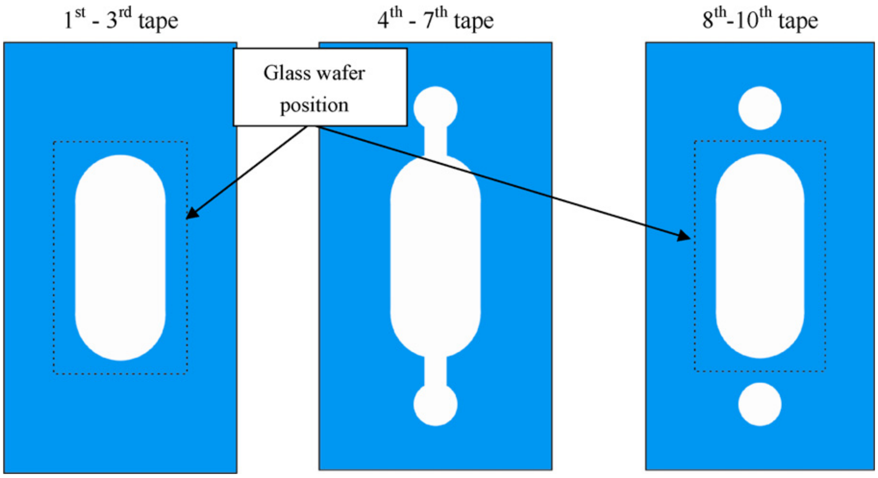


Fig. 9. The LTCC layers for the transparent microfluidic chamber.

Fig. 10. The LTCC–glass transparent microfluidic chamber.

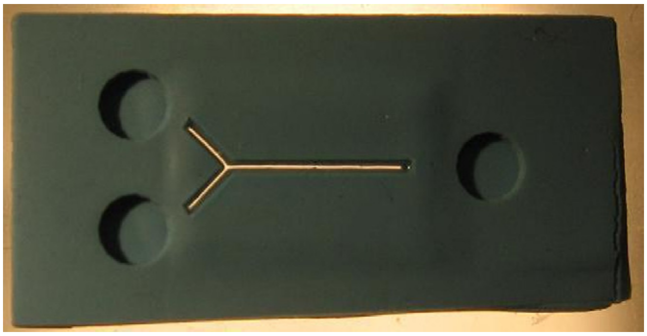


Fig. 12. Real picture of LTCC–glass mixer.

Fluorescence methods of analysis, which are commonly used in μTAS technology, often require a light excitation and an optical detection. Vertical and horizontal windows, situated inside a single structure, provide both easy laser excitation and optical detection. The LTCC–glass chip with both kinds of windows was constructed according to the previous description. The structure with coupled laser beam is demonstrated in Fig. 16.

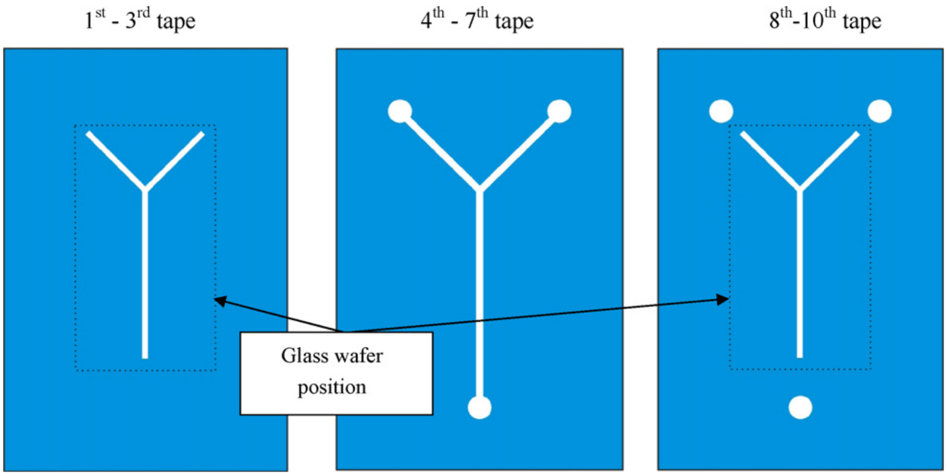


Fig. 11. The LTCC layers for the transparent microfluidic channels.

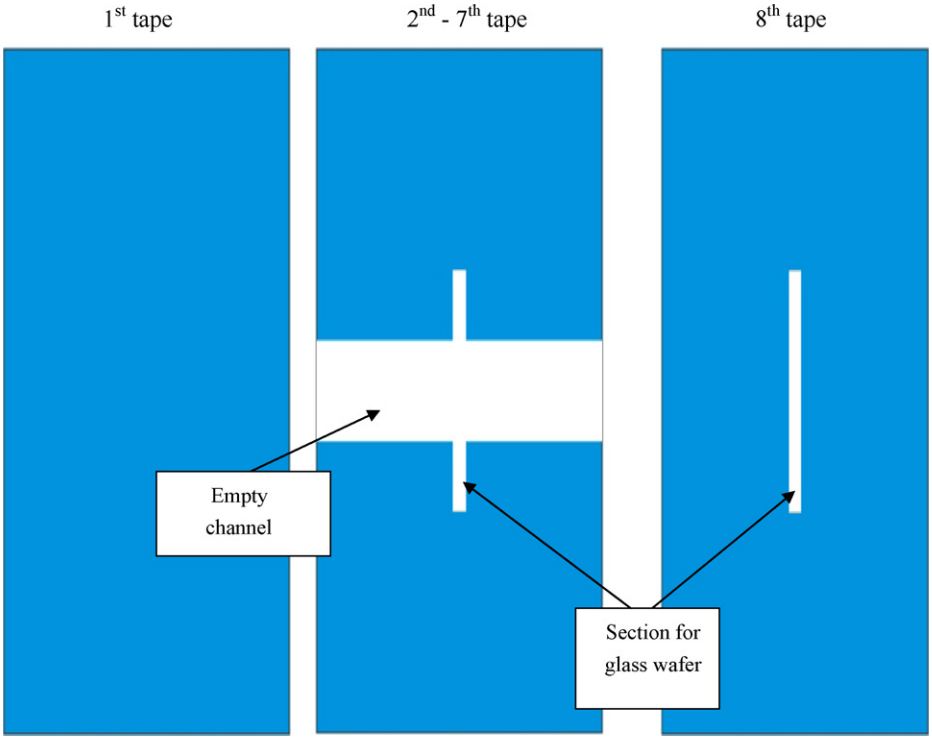


Fig. 13. The LTCC layers for the vertical window.

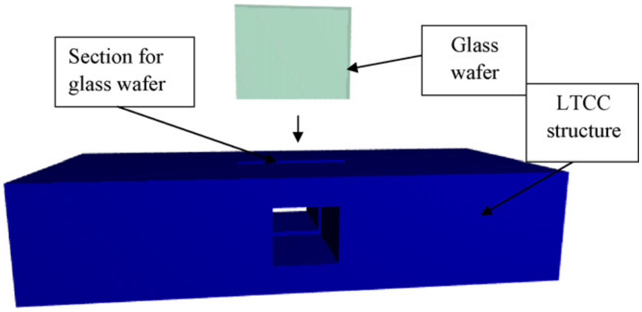


Fig. 14. The glass wafer location in the green tape structure.

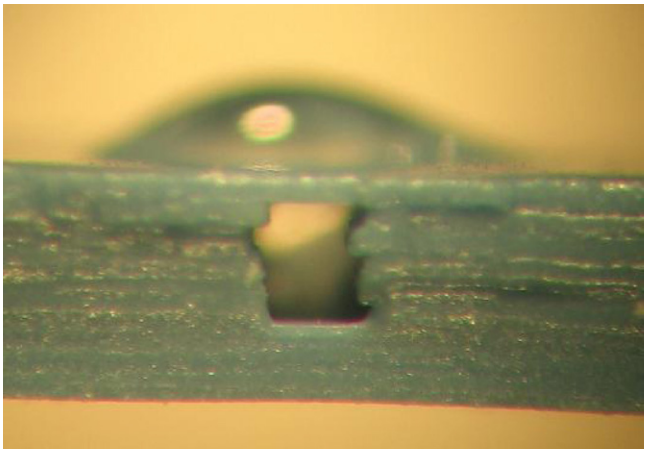


Fig. 15. Photo of the vertical window in the LTCC material.

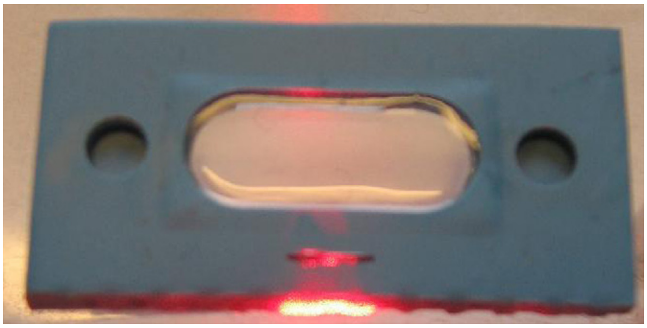


Fig. 16. Photo of the LTCC–glass structure with coupled laser beam.

7. Conclusion

The LTCC technology has been discriminated in μTAS application. The main reason has been non-transparency of ceramics. It has indicated problems with direct optical measurements. The presented novel technology enables ceramic structures to combine with transparent materials. The technique allows for the manufacture not only of horizontal but also vertical glass windows. Glass ports provide easy optical excitation and readouts. What is more, the technique is compatible with the LTCC technology and can be simply applied to standard processing. The microfluidic structures made of LTCC and glass are able to compete with silicon–glass elements in the μTAS application field.

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Paweł Bembnowicz was born in Poland in 1982. He was graduated from Faculty of Microsystems Electronics and Photonics in the Wrocław University of Technology in 2006. Currently he is a PhD student at Wroclaw University of Technology, where he is involved in designing and developing microsystems for biochemistry.

Leszek Golonka was born in Poland in 1946. He received his MSc and PhD degrees in electronics from the Wroclaw University of Technology, Poland in 1969 and 1976, respectively. In 1991, he received the DSc degree. Since 1996, he has been a professor at the Wroclaw University of Technology. His current research activity includes thick-film and Low Temperature Cofired Ceramics (LTCC) devices, sensors and microsystems. He is IMAPS, IEEE and PTTS (Polish Society for Sensor Technology) member.