



DESIGN AND APPLICATION OF LOW TEMPERATURE CO-FIRED CERAMIC SUBSTRATES FOR SENSORS IN ROAD VEHICLES

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Abstract. Nowadays low temperature co-fired ceramics (further on: LTCC) are used in sensor technology as sensors and actuators. Sensors and actuators are playing an important role in intelligent road vehicles. These sensors and actuators are built up by LTCC Microsystems. The processing of starch powder and polymer based on sacrificial layer for fabrication of microfluidic structures of LTCC is described in this paper. In order to determine the optimal lamination parameters the quality of the structure was examined for 30 different temperature-pressure-time adjustments. Samples were examined by scanning acoustic microscope to detect the subsurface delamination and internal inhomogeneities. The acoustic microscopic image of each sample from different lamination method was evaluated by image processing algorithm in MATLAB environment.

Keywords: LTCC, fluidic channel, lamination, scanning acoustic microscopy, ceramics, vehicle.

Introduction

The LTCC technology started about 30 years ago as one of the multichip modules (MCM) technologies used for fabrication of multilayer ceramic substrates (MCM-C). Due to its high reliability and relative low co-firing temperature it is still widely used in the industry. The LTCC circuits are suited for high frequency and temperature applications e.g. bluetooth, microwave and automotive systems (Smetana *et al.* 2007; Modes *et al.* 2003). The advantage of LTCC technology in road vehicles are high performance combined with low volume. The following kinds of LTCC sensors and actuators are made in LTCC microsystems:

- temperature sensor (Dziedzic, Golonka 2000; Zawada *et al.* 2000);
- pressure sensor (Golonka *et al.* 2000; Partsch *et al.* 2003);
- proximity sensor (Gongora-Rubio *et al.* 2001);
- gas and liquid flow sensor (Gongora-Rubio *et al.* 1999);
- gas sensors (Teterycz *et al.* 1998);
- and thermocouples (Golonka 2006).

The microfluidics applications in LTCC substrate require various structures. These structures can con-

tain pressure sensor (Khanna *et al.* 2004), flow sensor (Gongora-Rubio *et al.* 1999), force sensor (Birol *et al.* 2007) and gas sensor (Achmann *et al.* 2008). Combining and using these miniaturised system in road vehicles with integrated electronic modules, Micro Total Analysis Systems (μ TAS) and Micro Electro Mechanical Systems (MEMS) applications were realized (Ibañez-García *et al.* 2006). Such systems could be used to measure and control AFC (air fuel concentration).

The construction of these complex three-dimensional geometries of cavities and channel networks brought challenges to the research groups (Birol *et al.* 2005). The main advantage of LTCC-based microsystems containing embedded channel is that the multilayer substrate is the carrier of silicon components and combines good electrical properties and the capability of creating fluidic structures. Recently, the LTCC has become one of the most popular materials of microfabrication techniques for the production of three-dimensional structures beside silicon and polymers. The fabrication of LTCC microsystems is cheaper than silicon ones and quite fast (Golonka 2006), and the resolution of the structure is higher than a molding technique of rapid polymer-based microfluidics. Although the sizes

of the microstructures realised in silicon (about 1 μm) are smaller than in LTCC but such a small size is not required for every microfluidic application. Furthermore, microstructures in silicon are usually created by etching, therefore without adequate thickness it is not able to stand higher mechanical stress. Due to the relative larger size microstructure in LTCC has the advantage of simpler handling and it does not need expensive package (Gongora-Rubio *et al.* 2001). The technology provides the integration of the electronics, the non-electrical functions and package in one unit which is effectively utilized in the highly integrated LTCC based microfluidics. In addition, the LTCC substrate is processed in raw state, which condition brings closer the design and the fabrication (Thelemann *et al.* 2002).

Reliability also plays a very significant role especially in the automotive industry. Sometimes these applications have to stand high power, temperature or mechanical stress. Therefore, LTCC is an accepted material for these purposes. It is suitable for high-performance engine control unit (Nishigaki *et al.* 2004), gear control units, anti-blocking breaking systems, stability and speed control devices (Bechtold 2009) or fuel cell application (Partsch *et al.* 2006). In some cases the demand of complexity and reliability requires the integrated handling of fluid or gaseous substances (Schmid 2002; Schmid *et al.* 2006).

The typical process of the LTCC technology involves the steps: blanking the tape to the desired size, via formation by laser or mechanical punching, via filling with a (viafiller) conductor ink, deposition of the conductive and passive layers by screen printing method, stacking and laminating the layers and finally co-firing using the described heating profile. After co-firing additional conductors or passive component can be screen printed and placed on the top and bottom surfaces of the substrate (Golonka 2006). So the LTCC circuits consist of glass-ceramic layers, internal and external conductive and passive elements, vias and discrete component (chips, resistors, capacitors, inductors) (Golonka 2005). However, the realization of microsystem application requires the modification of the standard LTCC technology in the manufacturing process. The technological modifications apply not only to the parameters but further steps should also be inserted. In the fabrication of embedded structures in a LTCC platform the screen printing is followed by the pre-lamination of the bottom layers, after that the cavity is filled with sacrificial volume material (further on: SVM) before the final lamination and co-firing (Peterson *et al.* 2005). Especially the optimisation of lamination parameters and choice of SVM require attention because the standard lamination method would damage the embedded structure and the result will be undesired deformation. The realisation of these structures is difficult without an isostatic laminator. The applied pressure can also differ from the conventional pressure value. Lamination time and temperature can be also changed for successful channel realization.

This research deals with the channel fabrication for such microsystem applications. Frequently appeared

problem of this production is that the inner structures damage and sag due to lamination pressure. To decrease these deformation two types of SVM can be used. Temporary filler materials support the cover layers of the structure only during the lamination, after this step the filler material has to be removed. The other possibility is the use of SVM which evaporates or burns out during the firing process. One of the widely used SVM is the carbon because it burns out easily and leaves no contamination after firing although the occlusion of the LTCC pores before total SVM burnout can lead to damage of the structure. (Birol *et al.* 2005; Khoong *et al.* 2009). The choice of high-order alcohol, polysaccharide or polymer as SVM which has lower decomposition temperature can be the solution for this problem. These materials also burn out completely without residue.

The deformation of the cover LTCC layers over a cavity has been examined by Khoong in case of different lamination pressures, although the separation of the laminated layers was not investigated (Khoong *et al.* 2009). A number of different lamination approaches are in use. Low pressure lamination is applied by Malecha and Khoong which results in less deformation of the embedded cavity but delamination is possibly occurred (Khoong *et al.* 2009; Malecha, Golonka 2009). Short time lamination (2 min) with different pressure values was utilised by Balluch and Wang but the effect of lamination duration has not been investigated (Balluch *et al.* 2008). Cold chemical lamination method was developed by Jurkow (Jurkow *et al.* 2009), Golonka and Gongora have developed double step lamination to reduce the sag of the chambers (Gongora-Rubio *et al.* 2004; Malecha, Golonka 2008) while the others carried out the lamination process using the prevailing laminating parameters that are 200–300 atm at 70 °C for 10 minutes (Ibañez-García *et al.* 2006; Birol *et al.* 2005; Espinoza-Vallejos *et al.* 1998; Smetana *et al.* 2007, 2009). Applying high pressure lamination the adequate integration of the glass-ceramic layers can be achieved but channel distortion usually occurs (Malecha, Golonka 2008). The application of adhesives between the layers enables the reduction of the lamination pressure although the reliability of this type of bond and the effect of the chemical reaction between the LTCC tape and the adhesive has not been investigated yet. As the literature review shows (Khoong *et al.* 2010) many different lamination techniques have been already published: the lamination pressure varied from 2 MPa to 30 MPa, the applied dwell time was between 2 min and 10 min, the lamination temperature was in the range of 40 °C to 100 °C. However, it is impossible to give certain conclusion of the whole structure without comprehensive examination. In most experiments the cross-section of the specimens is investigated which gives an overview from only a region of it.

In this work two different SVM (corn starch and a UV-polymerized material) were applied. Series of lamination experiments were carried out for 30 different temperature-pressure-time adjustments which have an effect on the quality of the structure. The cross-sectional geometry of the channel was examined

(Wolter *et al.* 2005) and the resulting inner structures was analysed by scanning acoustic microscopy (SAM) extended with semi-automatic image processing algorithm, which has not been used as a verifying method for the lamination of LTCC containing embedded structure until now. Based on the results, using the optimal lamination parameters U-shape embedded channel with dimensions of 30×1×0.50 mm in LTCC substrate was fabricated.

1. Experiment

The manufacturing process of U-shaped channel test structures in LTCC substrate and the acoustic microscopic examination of the laminated structures are described in this section. The experimental method was proposed to analyse the effect of the different lamination schemes on the rate of the delaminated area and characterise the relevancy of these parameters.

1.1. Fabrication Process

The microfluidic test structure consists of three parts of LTCC structures, which was made from LTCC tape DuPont 951 (DuPont 2001) green tape (3×3 cm) and the thickness of the tape was 254 μm before firing. The fabrication flow diagram can be seen on Fig. 1.

Two layers in the bottom are base layers, the inner layers contain the fluidic channels, and the top ones have the channel outlets. The reason of the doubled layers with different functionality is to ensure the adequate mechanical properties of the complex structure. The cut of the raw substrate was performed by UV NdYAG laser equipment (Coherent AVIA 355-4500). Test structure containing U-shape channel was designed for the examination of lamination parameters. The structure has lengths of 30 mm, width of 1 mm and height of 0.50 mm as shown in the layout of the inner layers in Fig. 2. The following laser parameters were set on the machine for the cut preparation:

- average power: 4.1 W;
- pulse repetition frequency: 25 kHz;
- deflection speed of the beam: 10 mm/s;
- number of scans: 10.

The next step after laser cutting was the stacking of the glass-ceramic layers in the proper order. The bottom base layers and inner layers were initially pre-laminated using isostatic press (IL-4004 series, Pacific Trinetics Corporation, USA) with a pressure of 2 MPa for 10 minutes at the temperature of 70 °C. The deformation of the bottom layers was avoided by an aluminium plate with the thickness of 2 mm which was put under the stack during pre-lamination. At this lamination the upper cover layers were not laminated together with the inner and bottom layers in order to minimize the sag of the channel. Afterward the channel was filled up with polymer (FullCure 720) sacrificial volume material.

This process consisted of two steps. The structure of the SVM was designed in Autodesk Inventor mechanical design and 3D CAD software, and then the 3D Printing System (Objet Eden 250) finalized the printed layers by

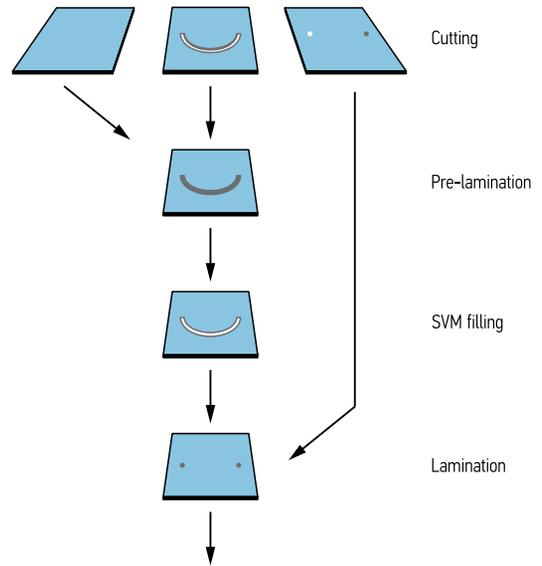


Fig. 1. Fabrication flow diagram of embedded channel in LTCC substrate (source: own edition)

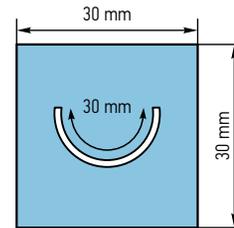


Fig. 2. Layout of two inner layers of the U-shape channel in the 3D LTCC module (source: own edition)

UV light, which polymerized the material. The lateral dimension accuracy of the production in case of this solid state SVM is 42 μm and 16 μm in the direction of thickness. The diameter of the laser beam of the applied laser machine is 20 μm thus the accuracy of the SVM production is in the range of the LTCC cutting accuracy. The polymerized SVM material was put into the cavity. After this process the top layers were laid on the LTCC laminate containing the filled channel and laminated for a second time. The temperature, pressure, and lamination time applied to LTCC with starch SVM is shown in Table 1. At the examination of lamination quality the SVM was starch because it has much larger residual deformation than polymer SVM and the aim is to decrease the sag of the channel without causing delamination. In our experiment we intended to use the changable parameters in the lamination process and the range was chosen according to the optimal data from literature (detailed in the introduction) and the suggested values of the manufacturer (DuPont 951 Green Tape data sheet (DuPont 2001)).

Finally, the substrate was burnt out. The heating rate was 2 °C/min up to 450 °C for the first period. After that the temperature was kept around 420 °C for 20 minutes. It was followed by a 9 °C/min heating period, until the temperature achieved 850 °C and kept it for 30 minutes.

Table 1. The optimal lamination parameters*

Lamination method [No.]	Temperature [°C]
1, 2, 3, 4, 5, 6, 7, 8, 9,10	40
11, 12, 13, 14, 15, 16, 17, 18, 19, 20	55
21, 22, 23, 24, 25, 26, 27, 28, 29, 30	70
	Pressure [MPa]
1, 2, 3, 4, 5, 11, 12, 13, 14, 15, 21, 22, 23, 24, 25	10
6, 7, 8, 9, 10, 16, 17, 18, 19, 20, 26, 27, 28, 29, 30	20
	Time [min]
1, 6, 11, 16, 21, 26	5
2, 7, 12, 17, 22, 27	7.5
3, 8, 13, 18, 23, 28	10
4, 9, 14, 19, 24, 29	12.5
5, 10, 15, 20, 25, 30	15

Note. *(temperature, pressure, time of lamination) in case of starch SVM were determined using different lamination methods (*source*: own edition).

In the course of this process organic ingredients evaporate from the LTCC, the SVM burns out, the glass matrix melts, coalesces in the substrate. During the last cooling down period the structure solidifies.

1.2. Examination of Laminated Structures by Acoustic Microscope

Scanning acoustic microscopy (further on: SAM) is very effective to determine the cracks and the delaminated areas in the LTCC stack, because it provides subsurface visualisation in a non-destructive way. The instrument uses a pulse echo method which means that the acoustic waves are reflecting from the discontinuity of substrate. Thus, echo is generated and reflected from a delaminated area which can be detected in the transducer. The lamination of tapes incorporating channels and cavities for the fabrication of microfluidic devices is a critical processing step. The reduction of the lamination pressure decreases the sag of channel, however, the probability of delamination is increasing. These delamination effects are usually not visible because they can be at the centre area of the substrate. By using SAM these delaminations can be also observed. Sonix HS1000 acoustic microscope was used to examine the samples. The set transducer frequency was 15 MHz and the scan speed was 164 mm/s. The frequency was relatively low in order to obtain information from the deeper layers as well. The acoustic images taken from different internal layer were evaluated.

The single reflection is described by the following equation (1):

$$R = \frac{Z_2 - Z_1}{Z_1 + Z_2}, \quad (1)$$

where: R is the reflecting factor [-]; Z_1 and Z_2 are the acoustic impedances of the materials realising the interface [$\text{kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$].

The acoustic impedance is determined by the density ρ and of the acoustic velocity v of the material (2):

$$Z = \rho \cdot v, \quad (2)$$

where: ρ density of the material [$\text{kg} \cdot \text{m}^{-3}$]; v acoustic velocity of the material [$\text{m} \cdot \text{s}^{-1}$].

The condition of the acoustic microscopic analysis is the difference between the acoustic impedance of the materials. The transmitting media was deionised water which has less than 10 times smaller acoustic impedance than the LTCC. In this case C-scan imaging mode was applied to examine the planes in different depth. The proper images from different depth are realised by a time scaled electronic gate which enables the evaluation of the echo only from one depth. The acoustic picture of each layer is taken by the transducer scanning over the sample.

The deviation of laminating pressure is different along the channel or cavity which leads to inhomogeneous densification of the LTCC structure. By increasing the lamination pressure the layers permanently bond together. On the other hand, it results in deformed and sagged structure or even crack formation. In contrast too low pressure is not enough to bond the layers together and causes separation of the layers. In addition, crack was observed on the cross sectional image around the channel outlet (Fig. 3.) in case of high lamination pressures and without suitable support, e.g. sacrificial volume material (SVM) filling.

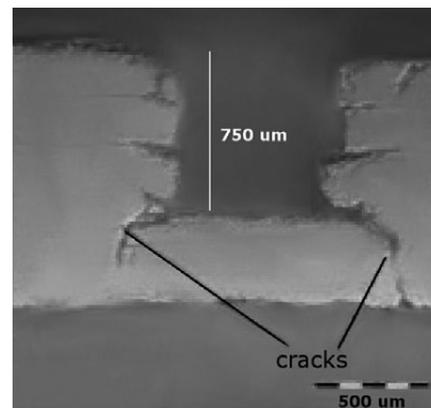


Fig. 3. Tearing effect can be observed around the channel outlet as a consequence of the inhomogeneous forces during lamination (*source*: own edition)

Only a few dents were noticed and several samples seemed intact. On the other hand, large subsurface defects could be observed by acoustic microscope at these samples. From the barriers strong shade difference was detected on the SAM images caused by large acoustic impedance changes. This investigational method was supplemented by image processing algorithm so it could be also integrated into a mass production line as inline characterization if the maximal delaminated area is given.

2. Results and Discussion

Analytical investigations have been carried out to obtain the optimal lamination parameters of LTCC with embedded channel elements. Channels were fabricated by 30 different lamination methods. The quality of each laminated structure was examined by scanning acoustic microscope. The subsurface defects can be detected by this non-destruction test and provides a complete scan of the multilayer structure. Experimental results show that with higher pressure and longer lamination time, the cover layers are more sagged so the channel is deformed in case of compressible filler material. Otherwise lower pressure value is not able to laminate the LTCC sheets together, which results in layer delamination. The advantage of polymer SVM is its much lower compressibility, and even in case of wide channel the desired shape dimensions of the 3D structure can be ensured. This polymer SVM designed in CAD is a breakthrough in this field, it is now the most precise method of realizing sag-free cavities due to the fact that it does not move during the lamination, does not enable channel deformation and provides accurate filling up. Moreover, the solid SVM penetration through multiple layers reduces their slipping which is a typical delamination effect.

A semi-automatic image processing algorithm was developed to analyze the delamination of the burnt LTCC substrate using the SAM images. The algorithm was written using the Image Processing Toolbox of MATLAB. Delamination defects are often concentrated to the inner layers of the substrate therefore it cannot be detected by simple visual testing. SAM inspection can be an ideal non-destructive testing method for delamination detection and also promotes the automation of the qualification process. The qualification method has applied two SAM images from the burnt LTCC substrate: one from the surface and the other from the inner layers (Figs 4 and 5).

The surface image is used to find the area of the microchannel because here the inner layer delamination has usually negligible effect. If this cannot be executed because e.g. delamination of upper layers the channel area can be given manually as the geometrical area of the channel. In case of delamination-free upper layers, first the grayscale image has to be binarised. Otsu's method (Sezgin, Sankur 2004) was used to determine the optimal threshold level for binarisation. The binarised image may contain areas that do not belong to the microchannel therefore they need to be removed. The algorithm searches for distinct 8-connected objects (Haralick, Shapiro 1992), and removes the ones whose size is less than a predefined level. At this point the only remaining black area on the image is the channel itself. The dilated channel area can be seen on Fig. 6.

The inner layer SAM image is processed in a similar way to the surface image. The algorithm searches for dark and bright areas using binarisation. After binarisation the closed objects are filled completely, because delamination always exists in a complete area. The microchannel area is subtracted from these areas, because



Fig. 4. SAM image taken from the surface (source: own edition)

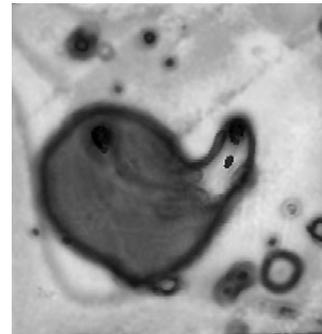


Fig. 5. SAM image taken from the inner layers (source: own edition)

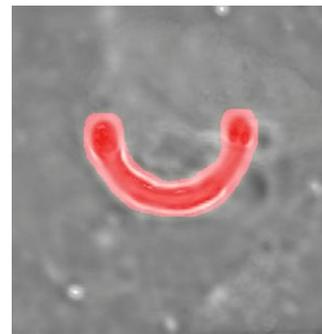


Fig. 6. The microchannel area on the LTCC substrate (source: own research)

there cannot be delamination. Fig. 7 shows the results of the image processing.

The white and the black coloured areas in Fig. 7c show delaminated sections. We can define a number – called delamination ratio (DR) – which is the delaminated area divided by the area of the substrate under examination (3):

$$DR = \frac{DA}{SA}, \quad (3)$$

where: DR is delamination ratio [-]; DA is the delaminated area [cm^2]; SA is the substrate area [cm^2].

The evaluation of the image process algorithm is shown in Table 2 which summarizes the mean and deviation values of delamination ratio at different conditions.

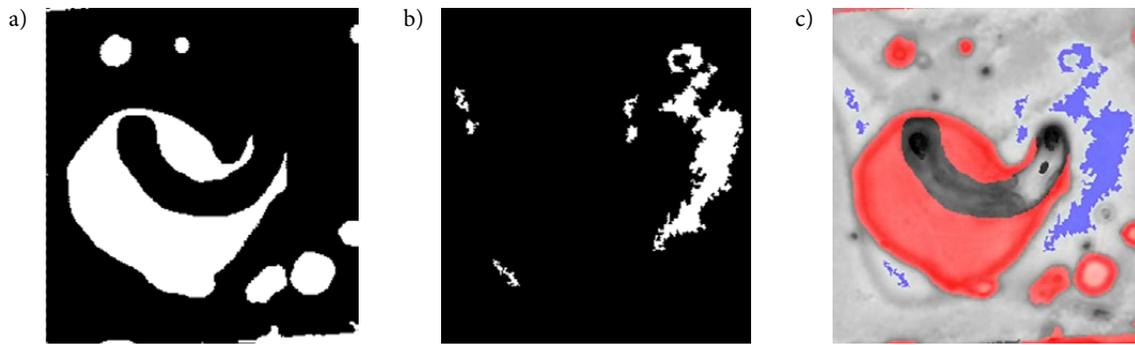


Fig. 7. Inner layer image analysis results for dark area (a), bright area (b) and the two combined (c) (source: own research)

Table 2. Distribution of delamination ratio grouped by lamination pressure, time and temperature

Pressure	Mean DR	DR deviation
10 MPa	34.00	11.90
20 MPa	5.50	4.76
Time		
5 min	21.50	17.80
7.5 min	20.50	18.02
10 min	18.00	18.23
12.5 min	19.33	12.93
15 min	19.17	10.75
Temperature		
40 °C	21.75	23.20
55 °C	20.00	20.15
70 °C	17.50	11.39

(source: own calculation)

In Fig. 8a the sample has been prepared with the 13th lamination method (55 °C, 10 MPa, 10 minutes). On the surface and at the upper layers of almost every sample minor unevenness problems can be observed. Between the inner layers of the stack large delamination was detected. The next sample (Fig. 8b) was produced at 55 °C and with the pressure of 20 MPa for 10 minutes. The sample has no significant delamination.

The sample in Fig. 9a has been prepared using the 21th lamination set (70 °C, 10 MPa and 5 minutes, see Table 1). Although there was not any damage on the surface, in deeper layers of the substrate delamination could be observed by SAM around the channel because of the lack of integration between the LTCC layers. The sample on Fig. 9b was made with duplicated 20 MPa pressure (26th lamination method). The sample did not have any distortion in the deeper layers.

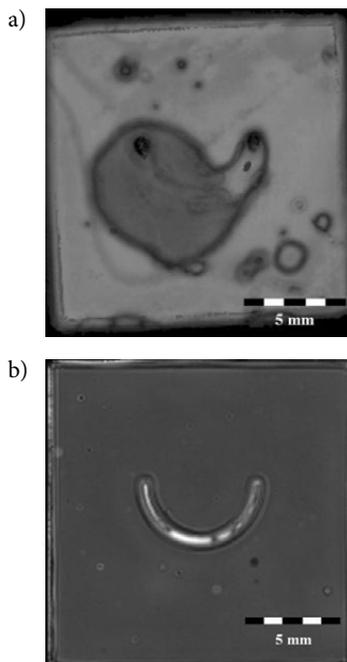


Fig. 8. On the SAM picture of the 13th lamination method large delamination can be observed (a); the 18th lamination method resulted in defect-free channel structure (b) (source: own edition)

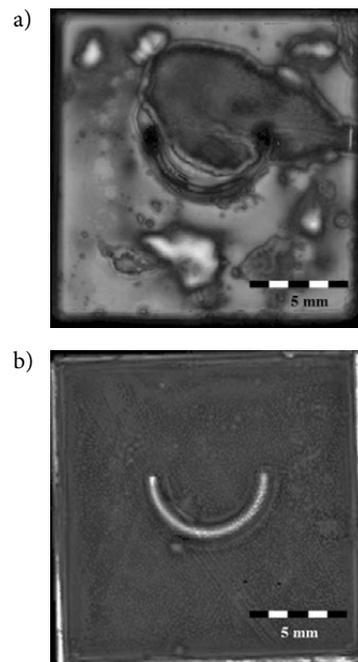


Fig. 9. On the SAM picture of the 21th lamination method the delaminations are caused by the inadequate pressure (a); the 26th lamination method laminated the layers permanently together (b) (source: own edition)

In comparison with samples made by different parameters it is concluded that the best samples of lamination were made by the application of the maximum 20 MPa because at lower pressure there is not enough binding force between the layers to keep them together. By increasing the lamination time there was no significant improvement observed. However, in case of longer lamination stronger channel sag was observed. Longer lamination time and higher temperature can ensure sufficient bond between the layers but the channel deformation is unacceptable. The use of solid state polymer SVM minimizes the rate of sag although it is not able to apply below 500 μm wide channel because in this size the material is fragile and tend to bend. Thus, the three technological factors cannot be universally defined, but design and the circumstances will provide the optimum combination.

Conclusion

Improved methods for realizing and verifying fluidic microchannel in LTCC substrate were presented. The optimal parameters for the isostatic lamination for LTCC substrate containing embedded channel were determined. The predesigned and formed solid state SVM is an absolute novel and innovative solution in the field of the multilayer LTCC formation technology and provides accurate channel geometry in case of wider channel as well. A verifying method of channel quality (delamination ratio) determination using scanning acoustic microscopy was developed by using image processing algorithm. The samples made by different parameters were examined by this method. It is concluded that the best samples of lamination were made, when the maximum 20 MPa pressure was applied. By increasing the lamination time there was no significant improvement observed but the channel sag has increased. In conclusion, to determine the optimal lamination parameters not only the reduction of channel deformation but also the appropriate binding between the layers have to be considered. SAM image processing algorithm is an ideal method to qualify the latter feature. An additional possibility could be the upgrading of the algorithm to a fully automated one.

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