

Impact of processing parameters on the LTCC channels geometry*

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A great advantage of Low Temperature Co-fired Ceramics (LTCC) yields the possibility of channel and air cavity fabrication. Such empty spaces have numerous applications, for example, in microfluidics, microwave techniques and integrated packaging. However, improper geometry of these structures can degrade the performance of the final device. The processing parameters recommended by the LTCC tape supplier are relevant for the production of multilayer circuits but not surface embedded channels and/or cavities. Thus, it is important to examine which factors of the fabrication process are the most significant. In our study, special attention has been paid to the geometric performance of the channel structure resulting from the applied processing parameters. Laser cutting parameters were checked to obtain the structures with great fidelity. The impact of an isostatic lamination on the quality of the final structure was analyzed. The influence of pressure and temperature of the lamination process on the channel geometry and tape shrinkage were examined. The performed experiments showed that some improvements in channel/cavity geometry may be achieved by optimizing the processing procedures. The microscopic observations combined with the Analysis of Variance (ANOVA) showed which combinations of the processing parameters are the best for achieving a channel/cavity structure with the desired geometry.

Keywords: *LTCC; channel fabrication; laser machining; lamination; DoE*

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1. Introduction

Low Temperature Co-fired Ceramics (LTCC) is considered to be a promising material for microsystems fabrication [1]. LTCC tapes are generally processed before firing (in green state). The layers with various electrical properties are deposited using the screen-printing technique. Some special structures can also be formed using mechanical milling [2], chemical etching [3], hot embossing [4] or laser cutting [5]. The LTCC tapes are stacked and laminated. These multilayer structures are then cofired. Finally, electrical elements can also be mounted, or the fired LTCC can then be bonded to different materials (e.g. silicon [6] or PDMS [7]).

Using these processes, the fabrication of sophisticated devices, for instance microfluidic devices for chemical [8] or biological [9] analyses, is possible. However, in order to achieve repeatable results, precise control over the fabricated devices is necessary. Moreover, LTCC tapes may differ in their composition (various organic binders and glass compositions are used). For each ceramic system, the processing conditions have a different impact on the quality of the final device.

In the present work, the impact of the parameters of different fabrication processes (laser cutting, lamination) of the LTCC substrates with incorporated microchannels has been studied. The LTCC tape that was tested was the Ceramtape GC material. This is a lead free material, so it is compatible with the RoHS directive. It has also been found that this tape is appropriate for the use in bio-applications [10–12] as well as high frequency

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applications [13]. These properties encourage the extensive investigation of this tape system.

2. Impact of laser cutting on processing fidelity

2.1. Experiment

We started our optimization with the laser cutting process. As it was reported in the literature, different sets of laser beam parameters should be used for various tape systems, depending upon composition, particularly the glass content [14–16]. The same laser equipment and incident beam have different impacts on various types of LTCC ceramics. According to these results, a theoretical model was proposed [17]. Also, some qualitative studies on different tape systems were presented [18]. We decided to use the Design of Experiment methodology to obtain quantitative information and a sufficient amount of experimental data for statistical analysis. This also enabled the investigation of interactions between input factors.

In our research, we analyzed four different inputs: the frequency of laser switching, laser beam velocity, and laser-on and laser-off delay. Every factor had three degrees of freedom. They are presented in Table 1. Our experiments were conducted using the LPKF ProtoLaser U, a Nd:YAG type laser, commonly used in the microelectronic industry. The laser operates at 355 nm with 4 W maximum power. The beam diameter in focus is 20 μm and repeatability is 2 μm , as given by the manufacturer in the datasheet. Ceramic material is removed by the ablation mechanism.

Four factors with three degrees of freedom give $3^4 = 81$ possible combinations. To reduce the number of necessary experiments, we used L9 orthogonal matrix. Thanks to this, the number of experiments decreased to nine. A combination of factors for each experiment is presented in Table 2. To minimize the influence of other, uncontrollable factors on the results (for example, ambient temperature), the experiments were performed in a random order. In each experiment, the sample was placed inside the processing area. Then, the beam

parameters were set according to the experiment matrix. Three test patterns were cut for each set of parameters.

We have chosen a Siemens star as a test pattern. It is commonly used for establishing process fidelity. Its simplified view is presented in Fig. 1. Our process benchmark was dimension D. The quality of the process was inversely proportional to the distance between opposite “edges” of the star. Thirty three measurements of the distance were taken for every star, which gave us 891 measurements, all done by means of a digital optical microscope. Examples of experiment results are presented in Fig. 2.

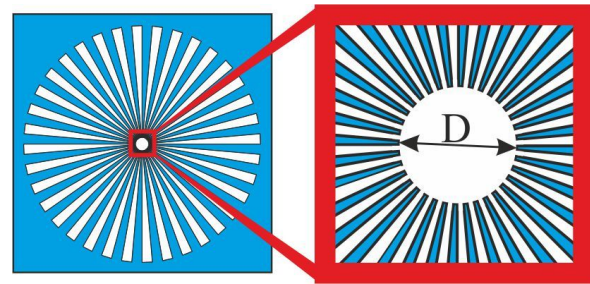


Fig. 1. Simplified figure of a Siemens star.

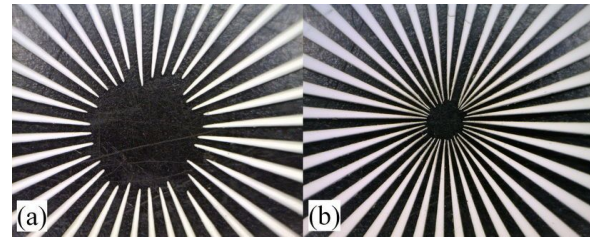


Fig. 2. Examples of a cut Siemens star: (a) experiment 1; (b) experiment 6.

2.2. Results

In Table 3, there are presented the values of mean distances between the opposite edges of the star as well as the standard deviations SD. The benchmark of process fidelity is the mean value: best results are obtained at the lowest values. On the other hand, standard deviation gives information about process robustness and pattern reproducibility. One may observe that the worst results

Table 1. Range of changes of analyzed inputs in laser cutting experiment.

Input	Abbreviation	Input level		
		Minimal (1)	Nominal (2)	Maximal (3)
Laser frequency [Hz]	f	40	70	100
Velocity [mm/s]	v	100	1300	2500
Laser-on delay [μ s]	t _{on}	0	50	100
Laser-off delay [μ s]	t _{off}	0	50	100

Table 2. L9 matrix used for the analysis.

Experiment	1	2	3	4	5	6	7	8	9
Level of factor f	1	1	1	2	2	2	3	3	3
Level of factor v	1	2	3	1	2	3	1	2	3
Level of factor t _{on}	1	2	3	2	3	1	3	1	2
Level of factor t _{off}	1	2	3	3	1	2	2	3	1

were obtained when there were no delays (red dashed frame). This could be caused by an inaccuracy of mechanical and optical system cooperation. Before the experiment, one may have expected that maximum delays would ensure stabilization of the whole system. However, the best patterns were obtained when both times were equal to 50 microseconds (green dotted frame). For longer periods, we observed fidelity or reproducibility degradation. A possible explanation can be derived from the amount of energy gathered in the resonant cavity of the laser. For longer periods of time between the “off” and “on” states, more energy stayed inside this cavity, and was released after switching the beam on. This resulted in higher power densities in the points that had been exposed to laser light.

The values of the mean distances with standard deviation bars for beam velocity and frequency are presented in Table 4. As can be seen, the worst stars were fabricated at the lowest beam velocity. On the other hand, the results obtained for beam velocities equal to 1300 mm/s and 2500 mm/s were comparable. Similar consideration of frequency as a variable did not lead to a clear conclusion. Generally, for the lowest switching frequency, the standard

deviation and mean value of the measured dimension were higher than those of higher frequencies. This was caused by the energy of laser pulses, depending on the switching frequency (higher frequency – lower energy of a single pulse). This made the process more susceptible to the change in material properties at certain points. The best geometry was obtained at a velocity of 1300 mm/s and frequency of 70 kHz.

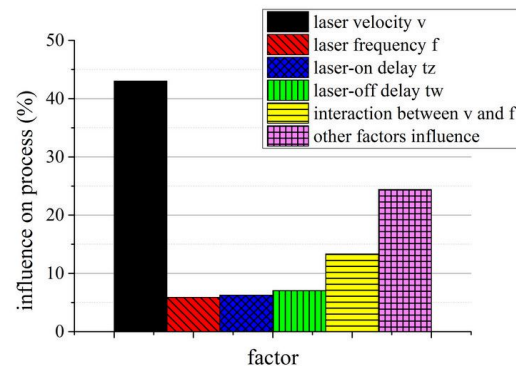


Fig. 3. Factor significance on process fidelity.

Finally, the Fischer-exact test was used for the evaluation of the factor significance. The results are presented in Fig. 3. As can be noticed, the most important factor was beam velocity (43 %). Laser-on and laser-off delay had an influence of 6.4 % and 7.1 %, respectively. The laser switching frequency had the smallest influence on this process, at a level of 5.9 %. However, the interaction between the beam velocity and laser switching frequency was very important. This is schematically explained in Fig. 4. When the frequency was too low for high velocities, then the beam hit the target only in

Table 3. Mean values and standard deviations for different laser-on and laser-off delays.

$t_{\text{off}} [\mu\text{s}] \backslash t_{\text{on}} [\mu\text{s}]$	0		50		100	
	mean [μm]	SD [μm]	mean [μm]	SD [μm]	mean [μm]	SD [μm]
0	254.5	142.9	51.8	39.9	41.8	9.2
50	48.2	12.8	56.2	13.5	87.5	29.4
100	63.6	42.7	212.4	70.8	59.6	16.7

Table 4. Mean values and standard deviations for different switching frequencies and beam velocities.

$v [\text{mm/s}] \backslash f [\text{kHz}]$	40		70		100	
	mean [μm]	SD [μm]	mean [μm]	SD [μm]	mean [μm]	SD [μm]
100	254.5	142.9	212.4	70.8	87.5	29.4
1300	56.2	13.5	41.8	9.2	63.6	43.7
2500	59.6	16.7	48.2	12.8	51.8	39.9

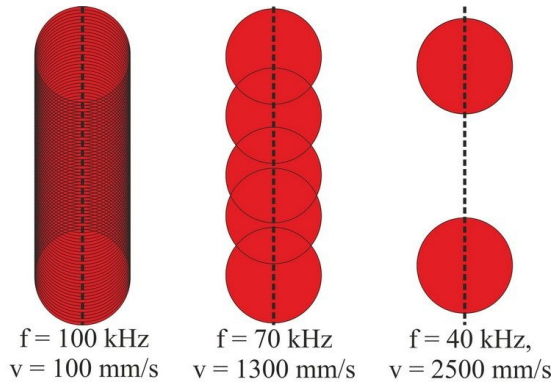


Fig. 4. Laser beam overlapping for different combinations of velocity and frequency.

a selected point, but with a greater pulse energy. Otherwise, the beam overlapped and cut the patterned shapes that were more uniform. Similar conclusions were drawn by other researchers [17, 18].

3. Impact of lamination parameters on channel geometry

3.1. Experiment

In order to investigate the influence of the lamination process on microchannel geometry, we

designed the test samples presented in Fig. 5. We placed five sets of channels on a substrate with dimensions of $50 \times 100 \text{ mm}^2$ (before firing). Each one consisted of five channels with different widths, equal to 0.1 mm, 0.25 mm, 0.5 mm, 1 mm and 5 mm (in a green state). The thickness of a single LTCC tape was 0.3 mm.

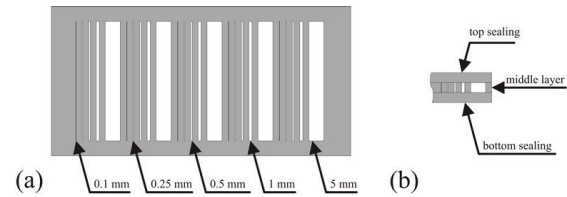


Fig. 5. Layout of the test sample: (a) middle layer with channels and (b) fragment of cross-section.

The test sample consisted of a 2-layer thick top sealing, 1, 2 and 3-layer thick channels in the middle, and a 2-layer thick bottom sealing (Fig. 5b). Firstly, the 2-layer thick pieces for the sealing of the top and bottom of the channels, plus the 2 and 3-layer thick pieces for the channels were fabricated. The lamination process was performed using the parameters recommended by the tape manufacturer (isostatic process in 70°C and 20 MPa). After the lamination process, the channels were cut in the

1, 2 and 3-layer thick middle layer. The channels were cut using the UV laser system (LPKF Proto-Laser U).

Afterwards, the experiments for the examination of the influence of three factors (pressure, temperature and channel height) on the microchannel geometry and LTCC structure shrinkage were designed (Table 5). To reduce the number of necessary experiments, (all combinations require 18) we used the L9 matrix. Input levels for each trial are presented in Table 6. Finally, all test structures were fired together using the thermal profile recommended by the tape manufacturer ($T_{\max} = 900\text{ }^{\circ}\text{C}$).

3.2. Results

After firing, the dimensions of the channels (height and width) were measured using a digital microscope. For each channel from a specific experiment, the width and the height were measured at 60 points, therefore, statistical analysis was used. The means and standard deviations were calculated. The benchmark defined error as the difference between the designed and measured mean value (lower value indicated better process conditions). Exemplary results for the 1 mm wide channel are presented in Fig. 6. One may observe that the best channel width was obtained with the factors combination A1 B2 C1, so the lamination conditions were $T = 25\text{ }^{\circ}\text{C}$, $p = 11\text{ MPa}$, at a channel height of one layer.

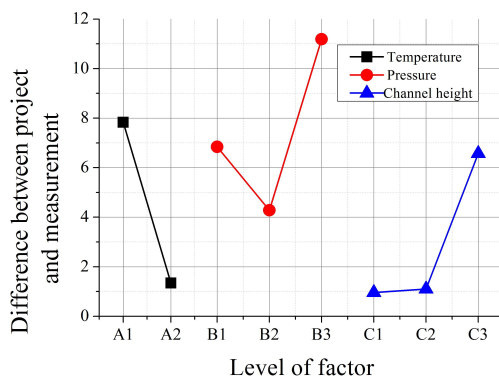


Fig. 6. Error of channel width for 1 mm wide channel.

In the next step, using the Analysis of Variance (ANOVA) methodology, we checked how strongly certain investigated factors (temperature, pressure and channel height) influenced the channel width and normalized height (height divided by the number of channel layers). The results are presented in Fig. 7. A value of 0 % means that this factor is statistically insignificant. To check this, the Fischer's exact test was used.

As can be seen in Fig. 7a, for the narrowest channel (100 μm), the width was independent of processing conditions, but the height was not. It is important to emphasize that smaller channels are more sensitive to measurement accuracy. Generally, it can be stated that for wider channels, pressure becomes more and more important, because pressure starts to act on larger surfaces of the channel walls. This is different for temperature and pressure. For channel height (Fig. 7b), one may observe that for the width up to 1 mm, the influence of pressure rises sharply and then remains stable (channels collapse). This has also been reported by other researchers [19]. For the finest structures, temperature is very important, because it causes softening of the organic binder.

From the point of view of process design, it is desirable to know the optimum parameters. This information is included in the pie charts in Fig. 8a, 8b and 8c. Usually, the fabricated channels were closer to the designed ones at a temperature of $25\text{ }^{\circ}\text{C}$ (Fig. 8a), which is not recommended by the LTCC tape manufacturer. In Fig. 8b, it can be seen that the lower pressure resulted in better geometry. However, these values must not be too low to prevent the delamination of LTCC layers. From the last chart (Fig. 8c), it was not possible to draw many conclusions, because the channel height was statistically significant in only 30 % of experiments.

To verify the conclusions obtained from the mathematical analysis, we made images of selected samples. We deposited thin aluminium film on the samples from experiment number 1 ($T = 70\text{ }^{\circ}\text{C}$, $p = 3\text{ MPa}$, $h = 1\text{ layer}$), 3 ($T = 70\text{ }^{\circ}\text{C}$, $p = 20\text{ MPa}$, $h = 3\text{ layers}$) and 8 ($T = 25\text{ }^{\circ}\text{C}$, $p = 11\text{ MPa}$, $h = 1\text{ layer}$) and inserted them under a Scanning Electron Microscope Hitachi SU6600. We observed

Table 5. Range of changes for analyzed inputs.

Input	Abbreviation	Input level		
		Minimal (1)	Nominal (2)	Maximal (3)
Temperature, T [°C]	A	25	70	—
Lamination pressure, p [MPa]	B	3	11	20
Height of the channel, h [layers]	C	1	2	3

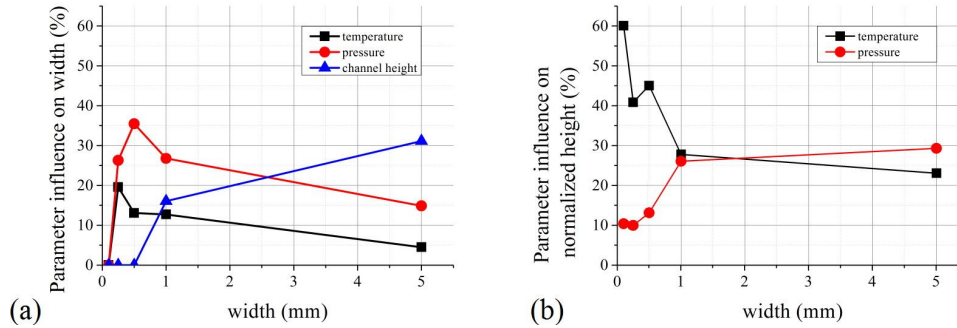


Fig. 7. Impact of different lamination parameters on: (a) width and (b) normalized height.

Table 6. L9 matrix used for statistical analysis.

Experiment	1	2	3	4	5	6	7	8	9
Factor A level	2	2	2	2	1	1	1	1	1
Factor B level	1	2	3	1	2	3	1	2	3
Factor C level	1	2	3	2	3	1	3	1	2

differences in channel geometry laminated at $p = 3$ MPa (Fig. 9) and $p = 20$ MPa (Fig. 10). One may notice, that higher pressure caused the rounding of the channel walls and corners. A channel fabricated at a lower temperature is presented in Fig. 11. As can be seen, the channel wall and its lower part are not connected. This proves that even if channel geometries are relatively better at lower temperatures, delamination of the layers may occur. This can result in fluid leakage from the channel, which can degrade the operation of the whole microfluidic system. In this case, the distance between the unconnected layers was even $30\text{ }\mu\text{m}$ (Fig. 12).

4. Impact of lamination parameters on shrinkage

4.1. Experiment

Our last experiment concerned the influence of lamination parameters on tape shrinkage in

the x, y and z axes. For that purpose, we fabricated the LTCC-based test samples. Firstly, we cut $50 \times 50\text{ mm}^2$ samples for lamination using a laser system. Next, we stacked six layers of the tape with distinct x and y axes. This was significant, because the LTCC tapes were fabricated in a tape casting process. As a result, the tape properties were different for perpendicular and parallel directions of the tape movement, and the manufacturer suggested two values of shrinkage. The laminations were conducted in the conditions presented in Table 7.

Table 7. Input values for different experiments.

Experiment	1	2	3	4	5	6
Temperature [°C]	20	20	20	70	70	70
Pressure [MPa]	3	11	20	3	11	20

After this, the laminated structures were cut into eight pieces with dimensions of $10 \times 20\text{ mm}^2$. For each piece, 4 measurements of length in x, y and z axes were taken. Then, all of the samples were fired using the thermal profile recommended by the LTCC manufacturer [10]. After that, the dimensions of the samples were again measured and the shrinkage was calculated.

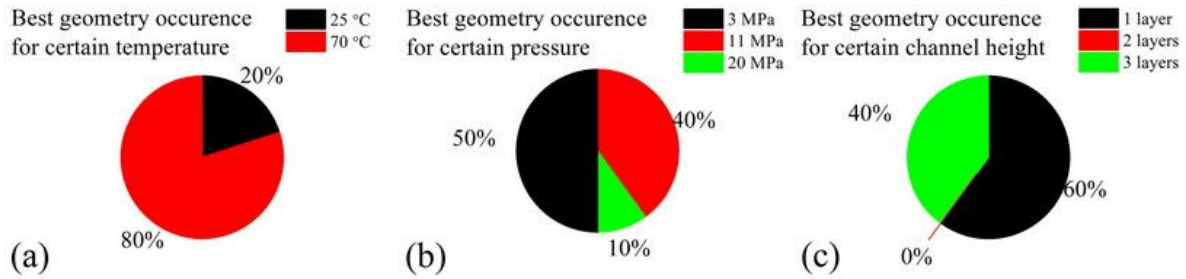


Fig. 8. Occurrence of the best geometry for different values of (a) temperature, (b) pressure and (c) channel height.

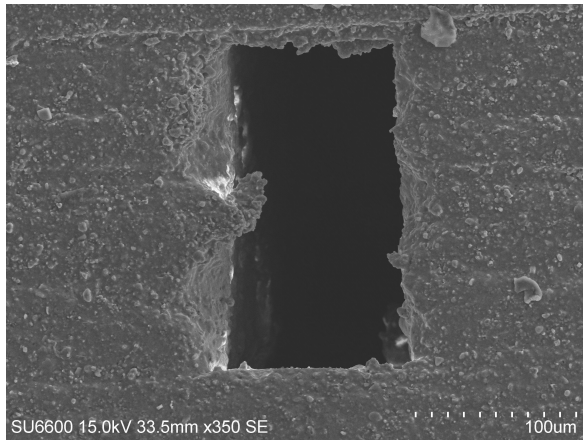


Fig. 9. SEM image of channel from experiment no. 1.

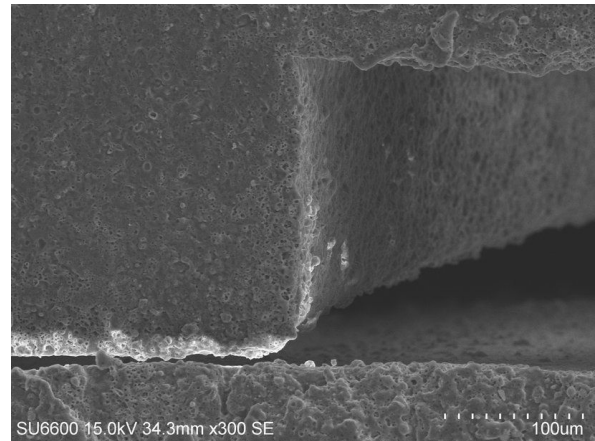


Fig. 11. SEM image of channel from experiment no. 8.

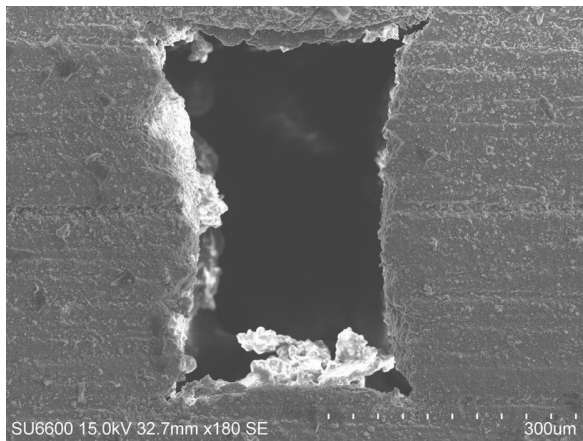


Fig. 10. SEM image of channel from experiment no. 3.

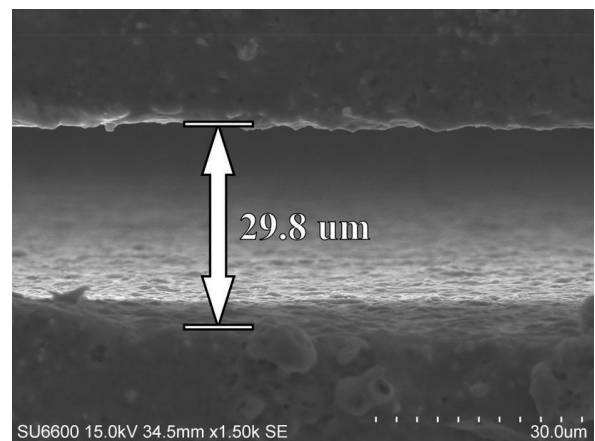


Fig. 12. Height of delamination.

4.2. Results

In Fig. 15, the factor significance, obtained from the Fischer's exact test is presented. Temperature has the highest influence on shrinkage. For the x, y and z axes they are 45 %, 63 % and

60 %, respectively. Next, there is a pressure, with x-shrinkage equal to 35 %, y-shrinkage of 26 % and z-shrinkage of 28 %. The influence of the interaction between those factors is about 8 % in x-axis and 10 % for y and z. One may observe that x

shrinkage is biased by a much higher error (12 %) than in the other cases (1.2 % and 2.1 %).

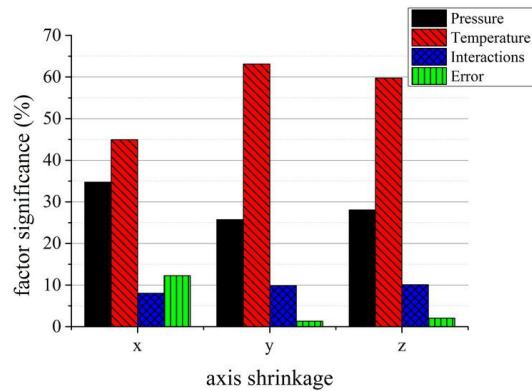


Fig. 13. Factor influence on different axis shrinkage.

In Fig. 16 and Fig. 17, the shrinkages in the x and y axes are presented. For the x-axis, the standard deviation is significantly higher than that in the y-axis. This corresponds well with the results obtained in the Fisher's exact test, where the error bar was also higher compared to that of other cases. For both axes, shrinkage decreased along with the increase of temperature and pressure. This was caused by the densification of the raw ceramic material. For x and y axes, the shrinkage values in the datasheet are given as 20.5 % and 21.5 % (± 1 %), respectively. In our experiment, the proximity to these values was obtained for the lowest values of temperature and pressure. The resulting data fit very well with the results presented in the literature [19, 20]. This suggests that the manufacturer measured shrinkage as a ratio between the structure dimension prior to lamination and after firing. If some spatial structures were cut after that process, their dimensions would depend on the lamination parameters.

The shrinkage in the z-axis was also investigated. The thickness of a single tape without backing tape, given in the datasheet, is 0.31 mm and that value was taken to calculate the shrinkage. The results are presented in Fig. 16. It can be noticed, that the plot trends are opposite to those in Fig. 14 and Fig. 15. This is a result of the fact that during the lamination, the structures were exposed to pressure and were squeezed. Therefore, some

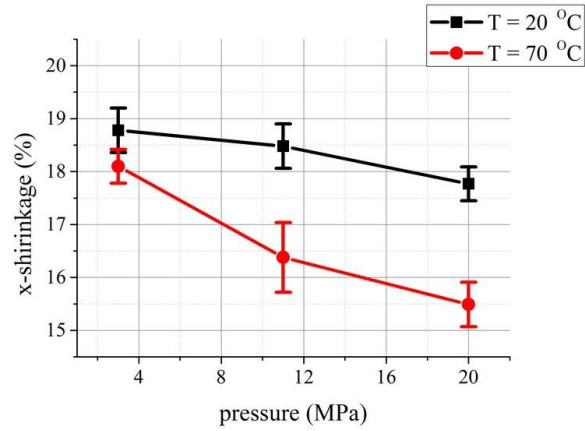


Fig. 14. Shrinkage in x-axis.

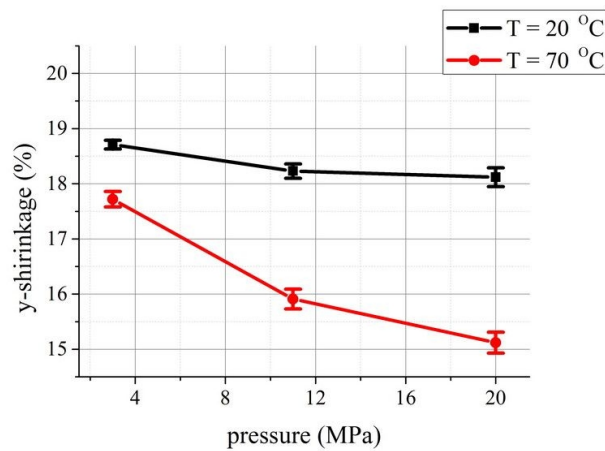


Fig. 15. Shrinkage in y-axis.

deformation occurred, which resulted in decreasing the thickness (Fig. 17).

5. Conclusions

Two stages of the LTCC fabrication process were examined. All experiments were performed using a Ceramtape GC LTCC material system. Firstly, we examined different sets of laser cutting parameters. We established that the beam velocity had the largest influence on fidelity. More important than the switching frequency was the relationship between frequency and beam velocity. Laser-on and -off delays were less influential, however, still affected the process. The influence of the lamination process parameters (pressure,

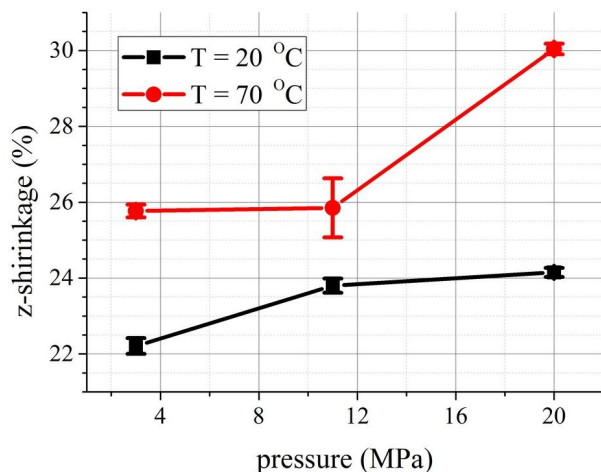


Fig. 16. Shrinkage in z-axis.

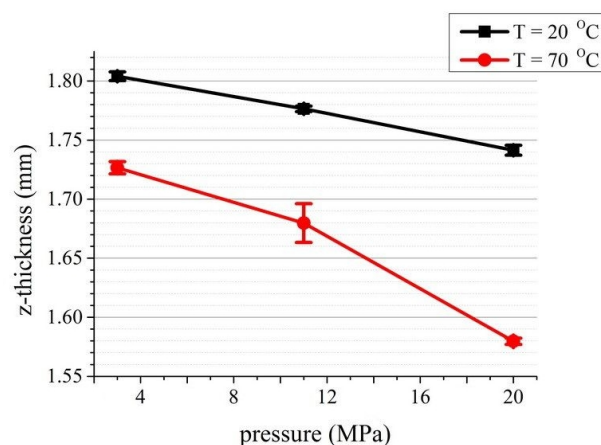


Fig. 17. Structure thickness after lamination process.

temperature) and the structural height on the quality of the microchannels embedded inside the LTCC multilayer substrate were examined. The combination of microscopic optical inspection with ANOVA analysis provided information about the significance of each factor. In order to verify the mathematical results we created images of the selected samples by means of SEM. This proved that at low temperature and pressure, the channels were less deformed, however, delamination could occur. Finally, we investigated how lamination parameters affected the shrinkage of the Ceramtape GC. We, established that with an increase in temperature and pressure, the

shrinkage decreased in the x and y axes, and increased in the z-axis. We suppose that it was caused by the tape densification and squeezing. The obtained results can be used for optimization of laser cutting and isostatic lamination parameters selection. This knowledge can be very useful in the development of multilayer LTCC structures, incorporating buried spatial structures for microfluidic and microwave devices.

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