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Precision fabrication of flexible microfluidic circuits using direct laser rapid prototyping solution

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Title:

Precision fabrication of flexible microfluidic circuits using direct laser rapid prototyping solution

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Abstract:

An effective development platform for custom lab-on-a-chip and lab-on-a-foil solutions has been regarded as a missing element for wider adoption of microfluidic technologies in everyday life. We have used a direct laser structuring device combined with CAD-CAM software and have developed an efficient, fast, and precise procedure for rapid prototyping of widely accessible contemporary materials utilized for flexible microfluidics. Utilization of an ultra-short pulsed laser has enabled us to predict and control the laser ablation process on thin low-temperature co-fired ceramics and Kapton foil. We have demonstrated an agreement between the theoretic predictions and experimental data on ablation rates, paving the way to a fully predictive manufacturing process. Our procedure enables an independent control of microfluidic channel shape, depth, and lateral dimensions down to 10 μm , while maintaining an exceptional process quality achieved by a parametrically optimized laser output at single-picosecond pulse durations. Our novel rapid prototyping solution features short turnover times, minimum material waste, no chemical procedures, and a single-step process free of heat-effects. We demonstrate the improvements to the structuring process on a known difficult-to-manufacture herringbone mixer structures inside a micro-mixer chip application.

Keywords:

lab-on-a-foil, laser processing, ultra-short pulses, microfluidics, micro-mixer

1. Introduction

Increasing interest of the microfluidics community in developing complete Lab-on-a-Chip solutions based on thin and flexible films, also called Lab-on-a-Foil solutions, has been recognized through recent developments¹. The manufacturing process needed to design, build, and operate the lab-on-a-foil systems is typically rather complex and involves chemical processes. While roll-to-roll manufacturing systems and similar have been proven to lower the manufacturing costs for large scale production², laser-based direct structuring approach has been recently proposed for classic microfluidic rapid prototyping applications³ due to short turn-over times and non-contact high precision manufacturing. Laser ablation by itself does not guarantee a high processing quality and thus optimization of processing parameters is necessary⁴, beginning with the choice of laser type and further optics, sample mounting, processing precision, software algorithms, etc. Processing precision and resolution are especially important in the field of microfluidics as imperfections lead to leaking, increased friction losses in the flow, and other unwanted phenomena^{5,6}.

Direct laser processing with ultra-short pulsed laser sources has received widespread attention in the scientific community. Within the microfluidics field, demonstrational use-cases have been presented on widely used classic microfluidic materials, such as glass⁷ and PMMA^{8–10}. Similar applications were also demonstrated on flexible materials, such as low-temperature co-fired ceramics (LTCC)^{4,11–13}, or for surface modifications of polyimides¹⁴ and PDMS¹⁵. Patterning of polyimide materials used for microfluidic applications is commonly achieved with reactive ion etches, primarily for point-of-care devices¹⁶, as Kapton is a biocompatible material^{17,18}, or even laser ablation¹⁹. Flexible materials are in general necessary for contemporary wearables²⁰, point-of-care applications²¹, studies of biological systems²², and other in-vivo applications.

While laser processing has already been considered as described above, we here demonstrate a highly optimized approach using a novel and versatile laser processing system. Optimized use of ultra-short pulsed lasers typically results in near-zero heat effects to the surrounding material, sparking the term ‘cold ablation’. On the other hand, the lack of optimization leads to the generation of thermal effects during processing. Ultra-short pulses with sufficient pulse energies are also able to activate nonlinear effects, thus leading to an increased precision of processing and enabling a wider range of applications. At the same time, using ultra-short pulses may offer an improved throughput of a laser-processing device. We used a laser-processing system capable of precisely tuning all the necessary parameters for a fully optimized laser ablation processing procedure. The system included a carefully chosen laser source, a precise mechanical table, sample position recognition, an advanced 2D galvanometric scanning system, and all necessary optical components for flat-field telecentric laser focusing.

In this work we present an optimized laser processing procedure using ultra-short laser pulses on flexible materials for microfluidic applications. We show that a clearly predictable material response follows from a simple laser ablation model that is applied to the experimental data. Decoupled control of channel width and depth is achieved through the application of this model. The final effects of structuring on the surrounding material were observed, with special attention paid to the possibly present heat effects to the surroundings, while also measuring the resolution, precision of the process, and the material’s response to the changing laser pulse energy. In the end, we show a direct comparison to recent work of other groups using similar materials for a micro-mixer chip, underlining the need to understand process of laser ablation in order to achieve the best possible outcome. The combined abilities to predict the processing and understanding of the underlying physical mechanisms pave the way for fully automated prototyping, thus opening our work to further upgrade and improve in the future.

2. Experimental setup

We used a commercially available direct laser microstructuring device ProtoLaser R (LPKF Laser & Electronics AG), based on a single-picosecond pulsed laser source. The device is specially designed for rapid prototyping applications with a high demand for precision and low thermal effects. It features a 1030 nm wavelength laser emitting approximately 1.0 ps long pulses at high repetition rates and laser power of up to 4.0 W. The laser is combined with a mechanical table system and a fast galvanometric scanning system that are altogether structurally decoupled from the effects of the environment.

The laser spot diameter in focus position of an f-theta lens is around 15 μm , allowing for the smallest line width of around 10 μm . We have used a combination of LPKF CircuitPro and LPKF CircuitMaster CAD/CAM software solutions to develop the optimal procedure for rapid prototyping of typical materials utilized for flexible microfluidics. Variations in scanning speed, laser repetition rate, laser spot size, and pulse energy result in different effective structuring speeds and pulse-to-pulse distance as a different number of passes is necessary. The same

parameters are reflected in the best achievable line/space distance that depends on the pulse energy used and becomes larger with an increasing energy. Typical processing parameters are gathered in Table 1, for both Kapton and LTCC materials. LTCC sheets were type SK-47 produced by KEKO Equipment, the same type as already used by other groups for microfluidic applications^{12,13}, and were $165 \mu\text{m} \pm 8 \mu\text{m}$ thick.

Parameter	Unit	Maximum	Typical	
			<i>Kapton</i>	<i>LTCC</i>
Scanning speed	mm/s	2000	400	2000
Pulse repetition frequency	kHz	200	40	40
Pulse energy	μJ	50	6-12	5-50
Effective structuring speed	mm/s	2000	6-400	20-400
Line/space repetition	μm	10/10	20/10	30/20
Pulse-to-pulse distance	μm	/	10	50

Table 1: Typical laser processing parameters.

Samples are held in place with a vacuum-table system that enables mounting and processing of samples as large as 300 mm by 200 mm while maintaining the resolution, structuring speed, and small structure size across the whole sample. The setup allows for consequent creation of multiple layers, i.e. for complete micro-reactor production⁴, in a single step with incorporated capabilities for cutting the samples, drilling through holes, channel engraving, or surface modification.

A laser-scanning microscope (Keyence VK-X 200) that can detect fine surface details and accurately measure the channel depth was used to characterize the structures. Additional imaging has been performed on an optical microscope combining transmitted, reflected, and dark-field illumination (Olympus BX53M) to achieve the best evaluation of surface properties after laser processing.

3. Calculations and results

Microstructuring experiments were performed with the goal of defining the best possible solution for a fast, reliable, and a quality-oriented rapid prototyping solution. We have considered different pulse-to-pulse distances on material, laser pulse energies, and different approaches to structuring, i.e. multiple scanner passes over a single line or over parallel lines to achieve different structure width and depth independently from each other and at an unperturbed surface quality.

First, we have evaluated the effects of an individual single-picosecond laser pulse on both chosen materials. Single pulses were always focused on the top material surface and their energy was varied for about two orders of magnitude. Further, we have tested a simple ultra-short pulse laser ablation model against our data on ablation processing of Kapton and LTCC sheets. The model predicts the ablation crater diameter d_c as a function of the pulse energy:

$$d_c(E_p) = \sqrt{2w_0^2 \ln\left(\frac{E_p}{E_{th}}\right)},$$

where w_0 is the laser beam waist radius, E_p is the pulse energy, and E_{th} the threshold energy for ablation. The expression was established by Furmanski et al.²³ by taking into account the Gaussian spatial intensity distribution of the pulse and basic ablation properties.

We have found a good agreement between the model and the experimental data as shown on the graphs in Figure 1 a using w_0 and E_{th} as free parameters for the fit. This agreement both confirms that we are working in the regime of ultra-short pulse laser ablation with negligible energy transfer to the surrounding material in the range of parameters used. At the same time, it enables us to decide on correct processing parameters and conceptually consider even parameters outside the range of the current processing system. A visual comparison of single-pulse craters at low fluence (just above the threshold) and at medium fluence within the optimal region is shown in Figure 1 b.

The same equations can be used to model the total ablated volume and further the ablation efficiency as a function of pulse energy used. It follows from the model assumptions described above that the ablation depth is a consequence of laser pulse fluence above the ablation threshold and the energy penetration depth (δ) in the material^{24,25}. Ablated volume of the model's parabolic ablation crater in this case equals to:

$$\Delta V = \frac{\pi \delta}{16 w_0^2} d_c^4 = \frac{\pi \delta w_0^2}{4} \ln^2 \left(\frac{E_p}{E_{th}} \right).$$

Ablated volume per pulse defines the lateral and transversal resolution of the micro-structuring process, but ablation efficiency needs to be considered in order to control the amount of residual energy transferred to the material surrounding the ablation zone and to optimize the overall processing through the choice of laser pulse parameters. We define the ablation efficiency η_{abl} as the ablated volume ΔV over the pulse energy E_p and considering the above equation we get the following expression for η_{abl} as a function of the pulse energy:

$$\eta_{abl}(E_p) = \frac{\Delta V}{E_p} \propto \frac{1}{E_p} \ln^2 \left(\frac{E_p}{E_{th}} \right).$$

On the other hand, as the ablated volume is proportional to the fourth power of crater diameter, we have measured it directly. Missing only a scaling parameter, a dimensionless (normalized) ablation efficiency can be calculated using the measured crater diameter and pulse energy as the following equation holds:

$$\eta_{abl}(E_p) \propto \frac{d_c^4}{E_p}$$

Plots of normalized ablation efficiency are shown in Figure 1 c and d for LTCC and Kapton, respectively. The normalization constant used is the same for both graphs, so the values can be compared directly.

The highest ablation efficiency can be calculated by deriving this equation and setting the derivative to zero. It follows that the highest ablation efficiency is achieved at $E_p(\eta_{MAX}) = e^2 E_{th}$ and the same holds for the laser pulse fluence. The relation has also been calculated and demonstrated by Neuenschwander et al.²⁶. The optimal range of fluences can be equally estimated from the data of either exact or normalized ablation efficiency data and corresponding best fits of model functions. Achieving a high ablation efficiency is important not only for high processing throughput but also to reach the desired consequence - lowest percentage of delivered laser energy is transformed into heat if this condition is met.

Figure 1: a) Ablation data with models, presenting crater diameter as a function of the pulse fluence. b) Dark-field technique images of low fluence regime craters (top) and medium fluence regime craters (bottom) on Kapton. Scale bars equal to 50 μm. c) and d) Ablation data with models, presenting ablation efficiency as a function of the pulse fluence for LTCC and Kapton, respectively.

We have both calculated the ablation threshold fluence values from the model used on graphs in Figure 1 a and measured the values directly. The directly measured ablation threshold fluence on Kapton was $0.6 \text{ J/cm}^2 \pm 0.1 \text{ J/cm}^2$, and the calculated one was $0.33 \text{ J/cm}^2 \pm 0.01 \text{ J/cm}^2$. Measured ablation threshold fluence on LTCC

material was $0.5 \text{ J/cm}^2 \pm 0.1 \text{ J/cm}^2$, and the calculated $0.66 \text{ J/cm}^2 \pm 0.07 \text{ J/cm}^2$. The discrepancy between calculated and measured results on LTCC material is negligible while not entirely so on Kapton. The reason may lie in the transparency of Kapton where the ablation process at low fluences starts through a different combination of linear and nonlinear absorption compared to higher fluences. The simple model used cannot describe such processes in details. Regardless of the physical processes involved, the model matches the data scaling rather well and enables a high degree of predictive powers above approx. $2E_{th}$. Figure 1 b shows low fluence regime single pulse craters, done with the laser fluence of 0.9 J/cm^2 , less than three-times above the calculated ablation threshold (0.33 J/cm^2) and medium fluence regime single pulse craters done at the laser fluence of 2.2 J/cm^2 , within the optimal fluence range for high ablation efficiency. We observed a radical increase in the modified area and ablated material volume, while additional debris or molten material ejection were not detected. The peak of the ablation efficiency graph gives us a range of the most efficient parameters, being at fluences between 1.5 J/cm^2 and 3.5 J/cm^2 for Kapton and 4.0 J/cm^2 and 7.0 J/cm^2 for LTCC. Outside of these windows, at low energies the structuring process gets increasingly slow and at high energies unwanted effects due to residual energy accumulation start to be more pronounced (i.e. melting and molten material ejection).

Figure 2 shows a comparison of various laser ablation processed channels on Kapton using single pass scanning at different laser fluences. Starting at a very high laser pulse fluence, residual heat related effects (seen as splashed in Figure 2 a through c) diminish as the laser fluence is reduced towards the most efficient ablation (Figure 2 d). As the total delivered energy also gets lower, a further decrease of laser fluence does not lead to an increased residual heating compared to the optimal conditions. Instead, the volume of removed material further diminishes, as it is already visible from the comparison in Figure 2 where channel width and depth decreases with decreasing fluence. Depth decrease trend is visible as the bottom of the channel gradually becomes in-focus together with the unprocessed surface.

To demonstrate the advantages of our optimized approach to laser ablation processing, we have structured channels

Figure 2: Laser ablation processing of Kapton using single pass scanning at different laser fluences: a) very high fluence - 12.8 J/cm^2 , b) high fluence - 6.4 J/cm^2 , c) upper part of the optimal fluence range - 3.2 J/cm^2 , and d) lower part of the optimal range - 1.6 J/cm^2 . Scale bars equal to $50 \mu\text{m}$.

with simultaneously defined depth and width (Figure 3). The results are precise to a single-micrometer level, with measurements showing maximum deviations below $2 \mu\text{m}$ (Figure 3 a and b). Those were demonstrated on channels either $50 \mu\text{m}$ or $100 \mu\text{m}$ wide, both with a target depth of $50 \mu\text{m}$. Channel width is defined through the number of parallel contours, while the channel depth is related to the number of scanning repetitions done. The known response of the material to a single laser pulse (Figure 1) helps to estimate the processing parameter window, shortening the time needed and ensuring highest possible structure quality.

Figure 3: a) The depth profile of a 50 μm wide channel designed to be 50 μm deep. Actual measured depth of 49.9 μm using laser scanning microscopy. b) The depth profile of a 100 μm wide channel designed to be 50 μm deep. Actual measured depth of 48.5 μm using laser scanning microscopy. c) The channel measured in graph a), optical microscopy image, scale bar equals to 100 μm . d) Demonstrational densely packed microfluidic circuit with an inlet on bottom right and a spiralling channel with width/separation dimensions of approximately 30 μm /20 μm respectively. Optical microscopy image, scale bar equals to 100 μm .

Structuring of arbitrarily complex channels is also possible, for example spiralling channels with inlets and outlets. This outline has enabled us to demonstrate closely packed channels with width and inter-channel separation dimensions of approximately 30 μm and 20 μm respectively (Figure 3 d). The goal was to keep the channel's aspect ratio to about 1:1.

Finally, we have used our setup and the optimized laser processing procedures to demonstrate its capabilities on known difficult-to-manufacture structures from a proven and published application – mixers in micro-reactor chips⁴. We have taken the approximate shape and exact size of herringbone mixer structures used as developed, manufactured, and used by Jiang et al.⁴. The channel and separation widths were designed to be 100 μm , structure height 500 μm . The results obtained using our optimized approach are shown in Figure 4 a, while the original result reprints are shown in Figure 4 b. Jiang et al. used two different LTCC material, HeraLock 2000 (HL2000) by Heraeus and GreenTape 951 by Dupont (DP951). The original CAD drawings presented in that paper reveal that wall and cavity sizes were designed to be of the same size, which is achieved also by our optimized approach, but not in the original structures. In addition to the size misrepresentation, the original structures feature irregularities at inner corners as well as some carbonization. The same cannot be found when using our approach. While Jiang et al. show that the structures are sufficiently well made for the application intended and need no further optimization for the application of performing harsh chemical reactions; we demonstrate the possibility to satisfy the requirements of applications needing a much higher precision of processing. The material used in our experiments (KEKO SK-47) is comparable in mechanical and electrical properties (according to manufacturer's datasheets and other research²⁷) and is also compatible with the more widely used DP951, while HL2000 differs in both structure and composition.

For successful implementation of laser processed flexible materials into system-level microfluidic devices, complete with inlets and outlets, tightness to liquid pressure, and the necessary large area uniformity, it is important

Figure 4: Comparison of LTCC processed a) on our experimental setup using SK-47 material and b) done by Jiang *et al.*, using either LTCC type HL2000 (top) or DP951 (bottom). Reprinted with permission from the Royal Society of Chemistry. Our resulting structures have an even distribution of wall and space dimensions, as was originally intended for all cases. We observed a near-zero heat affected zone and no detectable carbonization.

that manufacturing steps do not alter the original surface properties and overall material's integrity. We have used a Form Talysurf Series 2 stylus profilometer (by Taylor-Hobson Ltd.) to measure the surface roughness with 3 nm resolution perpendicularly to the surface, and 1 μm resolution laterally. We have measured the surface in the direct vicinity of the laser-structured microfluidic channels on both Kapton and LTCC materials, as well as virgin surfaces of both materials. The results span both 2D surface topography maps and surface roughness measurements (Figure 5), with output parameters being the arithmetic mean height (S_a) and maximum height (S_z). Both parameters were also measured on control areas of different non-processed samples, serving as control values. Wrap-up of S_a and S_z parameter values is given in Table 2. No conclusive trend of roughness change was observed, leading to a conclusion that ultra-short laser processing does not affect the properties of surrounding surface areas. Control values on Kapton even significantly exceed the surface roughness near laser-processed areas, indicating variations in material itself.

Surface roughness parameters (μm)	Kapton	LTCC
S_a – virgin (P1 area)	0.059	0.233
S_a – control area	0.126	0.232
S_a – (P2-P5 areas)	0.044-0.060	0.233-0.320
S_z – virgin (P1 area)	1.1	2.14
S_z – control area	1.36	2.62
S_z – (P2-P5 areas)	0.58 – 0.83	2.16-2.77

Table 2: Surface roughness parameters for processed and non-processed areas of Kapton and LTCC materials.

Processing of Kapton results in a small ridge surrounding the channel, measuring 200-500 nm in height. This is less than half the maximum height variation (S_z parameter) measured for both the control area and the P1 area of Kapton (Table 2). There is no ridge present after processing of the LTCC, which can be explained by fundamentally different material compositions – Kapton being a polymeric material, while LTCC being a granular ceramic. No changes to the overall material thickness or integrity were observed in the measurements.

Figure 5: a) Surface topography measurement on Kapton. b) Detailed views of Kapton surface roughness for selected areas – P1 and P4. c) Surface topography measurement on LTCC. b) Detailed views of LTCC surface roughness for selected areas – P1 and P4.

4. Discussion and conclusion

The ablation data presented on graphs in Figure 2 falls in line with model curves very well. This match gives an ability to predict the effects of laser ablation on the material, opening an opportunity to fully optimize the laser ablation manufacturing procedure with regard to the specific application requirements. Optimization of laser parameters leads to structures featuring smooth edges, well-defined corners, dimensional stability, correct scaling, and depth control. At the same time optimization is necessary to avoid generating heat related effects as ultra-short laser pulses do not guarantee that by themselves.

Our optimized processing procedure was applied to microfluidic circuits manufacturing, on a notable example of known difficult-to-manufacture herringbone mixer structures. The results show the above-mentioned features, including no carbonization and no detectable heat-related effects due to laser processing. We used optical observations and processed material measurements to confirm near-zero heat effects to the surroundings, very high precision, clearly predictable material response to the changing laser pulse energy, and all general advantages of direct laser processing. Further surface topography measurements show no measurable changes to material surfaces, except for a small ridge surrounding the channels in Kapton. We have thus demonstrated a single-step process capable of ultra-high quality prototyping for microfluidic applications on in-demand flexible materials. Further research is necessary to confirm that the optimized laser processing approach works on the full spectrum of different LTCC substrates; our preliminary research on DuPont materials points in a positive direction.

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Author Contributions statement

JM wrote the main manuscript text, prepared figures, and conceptualized the work. AM contributed to the data acquisition and analysis. JM and BP designed the work. BP and RP drafted the work and revised it. All authors reviewed the manuscript.

Additional Information

The authors declare no competing interests.