Fabrication of millimeter-long structures in sapphire using femtosecond infrared laser pulses and selective etching

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A B S T R A C T

This paper analyzes laser and etching parameters to fabricate open and continuous microchannels and stacks of such microchannels in the bulk of crystalline sapphire (α-Al₂O₃). The structures are produced using a two-step method consisting of laser irradiation and selective etching. Infrared femtosecond laser pulses are focused in the bulk to locally render the crystalline material into amorphous. The amorphous material is, then, selectively etched in hydrofluoric acid. Amorphous sapphire shows a high etching selectivity in comparison to its crystalline state, which makes this material very attractive for a use with this technique. However, some of its properties make the processing challenging, especially during the laser-induced amorphization phase. This paper studies the effect of laser parameters by a step-by-step approach to fabricate long structures (longest dimensions up to millimeters) of different shapes inside the bulk of sapphire. The minimum cross-sectional dimensions of the resulting structures (microchannels) vary from few hundreds of nanometers for the smallest channels to tens of micrometers for the largest stacks of microchannels. The effect of the variation of repetition rate, pulse energy and channel-to-channel distance on the microchannels and stacks of microchannels is studied. SEM micrographs of polished cross-sections are used to perform a quantitative and qualitative analysis of the morphology of the structures after laser irradiation and, subsequently, after selective wet chemical etching.

1. Introduction

Crystalline sapphire (α-Al₂O₃) is nowadays used as construction component or base material in many sectors of science and technology. The hardness of sapphire (9 on the Mohs scale [1]) and its transparency in the visible spectrum (from 450 nm to 2000 nm [2]), together with other physical and chemical properties make the material suitable in many applications in the fields of semiconductors (particularly with high efficiency Gallium Nitride LEDs [3–10]), and in photonics in general [11–14].

Processing of sapphire has been demonstrated using different methods: direct laser writing [15,16], mechanical sawing [17], dry (plasma) etching [18–20] and wet etching [21–23]. In this manuscript we study a two-step method consisting of laser irradiation of crystalline sapphire with subsequent modification of the exposed material into amorphized material and successive selective removal of the latter by wet etching.

Due to the transparency of sapphire, the laser beam can be focused inside the bulk. If femtosecond or picosecond laser pulses are used with intensities in the order of 10¹³–10¹⁴ W/cm² [24–26] absorbed laser energy [27] leads to the amorphization of the crystalline sapphire. Amorphous sapphire is selectively etchable by hydrofluoric acid (HF) at a 10⁵ faster rate than crystalline [24–26,28–31]. If the material is exposed to several, overlapping laser pulses, it is possible to create regions and volumes of amorphized material.

Fig. 1(a) shows a cross-sectional model of a single microchannel in the bulk of sapphire, which forms the targeted basic shape for this study. Such structures can be exploited for, e.g. microfluidic devices– in the form of millimeter long hollow microchannels. More complex shapes can be created by superposition of microchannels.

However, when laser pulses are geometrically overlapping with the aim to form large amorphized structures – such as one-dimensional modified lines (made by amorphous material, before etching)– a series of phenomena may affect the formation and morphology of the amorphous sapphire. The latter, in turn, affects the solubility of the formed material.

In fact, publications on etched channels in sapphire, often report cross-sections in which, after the wet chemical etching, the obtained structure is not completely hollow [14,24,25,27,33]. That is, in these structures hollow/open regions, where amorphized sapphire is dissolved, can be found, as well as crystalline/unetched regions. These latter regions can be characterized by series of parallel nanochannels, see Fig. 1(b), or by a discontinued and irregular structures Fig. 1(c). In 2008, Juodkazis et al. [32] studied this phenomenon and showed that overlapping single pulse modifications causes recrystallization of the amorphized material, which makes it non-etchable by an acid like HF. On the other hand, Gottmann et al. [24] showed the change in morphology of cross-sections of channels obtained with this method, and found that the results of laser irradiation and wet etching depends mainly on laser parameters and focusing conditions. More specifically, the authors showed that, within a

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range of parameters (mainly numerical aperture and energy per pulse) the cross-section of channels either show a series of amorphized parallel nanochannels (Fig. 1(b)), or hollow channels (Fig. 1(a)).

Both Juodkazis et al. [32] and Gottmann et al. [24] studied the effect of only a limited number of processing parameters. Despite these results, until now, a well-ordered study regarding the main factors playing a role on the final morphology of the irradiated lines and other structures (formed by overlapping lines) in the bulk of sapphire is lacking in the literature. A wider investigation is needed, in fact, to understand the problem and have a general view of which specific conditions determine the final shape and appearance and how to tune the settings to obtain exact and distinct types of channels.

2. Experimental set-up and analysis tools

Fig. 2 shows a schematic of the set-up used for the laser irradiation experiments presented in this work.

A KMLabs Y-Fi femtosecond laser source was used, which emits a linearly polarized laser beam at a central wavelength of $\lambda = 1030$ nm. The pulse duration of this source is 230 fs, measured with an autocorrelator (APE Berlin Pulse Check, Germany). Heat transmission phenomena inside the lattice of sapphire occur on a time scale in the order of tens of picoseconds [34]. Hence, the selected ultrashort pulse duration limits the heat transmission inside the sample during the laser pulse. The spatial distribution of the laser beam is nearly Gaussian ($M^2 < 1.2$).

Since the laser source does not offer the option of changing the repetition rate to less than 1 MHz, an electro-optic modulator (EOM, Model 360–80 by Conoptics, USA) is mounted after the laser source for pulse picking. A beam attenuator (Ultrafast Version, Altechna, Lithuania) is used to set the pulse energy of the laser pulses. A microscope objective (11,101,666, Leica Microsystems, Germany, NA = 0.7) is used to focus the beam to a spot of about 0.9 $\mu$m diameter (calculated). The microscope objective is mounted on a linear stage (ATS100, Aerotech USA), which moves the lens in the $z$-direction, allowing the focal spot of the laser beam to be positioned relative to the surface of the sample. The sample is fixed with a vacuum chuck on xy-stages (ALS130-150, Aerotech USA). The microscope objective is also used as magnifying element for imaging; that is, the light reflected by the sample passes again through the objective and a tube lens to arrive finally on a CMOS camera (DCC1545, Thorlabs, USA). This alignment facilitates the positioning of the focal spot in the bulk ($z$-direction) of the sample as well as in the xy-plane. It also allows to observe the progress and quality of the irradiation during machining.

After irradiation, the samples are inspected using a Keyence VHX 5000 (Japan) microscope for optical and polarized microscopy, as well as using a Scanning Electron Microscope (SEM, JEOL JSM 7200F, Japan). After HF-etching, samples are analyzed at the same positions as after irradiation.

3. Materials and methods

3.1. Materials

Circular sapphire wafers (2 inches in diameter) with a thickness of 430 $\mu$m and crystal orientation (0001), purchased from Crystech GmbH (Germany), were used. The circular samples were cut into rectangularly shaped (of various sizes) strips for easier handling.

The wet chemical etchant, hydrofluoric acid aqueous solution 50% (BASF, Germany) was used at room temperature.

3.2. Methods

To investigate one directional structures, the sample is moved in one direction ($x$ or $y$ in Fig. 3), while exposing the sample to single femtosecond laser. This results in a modified line/track, see Fig. 3(a).

The laser beam is focused inside the bulk of sapphire at about 50 $\mu$m below the top surface of a specimen. The velocity $v$ of the stage is kept constant at 1 mm/s. By varying the pulse repetition rate of the laser source, the geometrical overlap between the laser pulses can be varied.

In order to produce stacks of lines, as is shown in Fig. 3(b), the approach for single lines is applied repeatedly. That is, stacks of lines are produced by overlapping single modified parallel tracks. The geometrical overlap of adjacent tracks is limited by the minimal incremental step of the $xy$ stage, which equals about 50 nm. Since the smallest cross-sectional dimension is about 600 $\mu$m, it is possible to vary the lateral geometrical overlap between adjacent lines from 90% down to separated lines (no overlap).

The geometrical overlap is defined as the percentage of the overlap of diameters of adjacent pulses and is calculated as:

$$\text{OL} = \left(1 - \frac{\nu}{f \cdot B}\right) \times 100\%$$

Where $\nu$ is the scanning speed of the XY stage, $f$ the pulse repetition rate of the laser source and $D$ is the (calculated) diameter of the focal spot.

All the line-based structures are irradiated close to an edge of the specimen for easy inspection. Table 1 provides an overview of the range of the other laser parameters.
Cross-sections of the structures were obtained by grinding the sample along the direction shown in Fig. 4, and removing enough sapphire to expose the amorphous material. Subsequently, for ease of inspection of the cross-sections, the sample was polished to optical quality (average roughness $R_a < 5 \text{ nm}$). Grinding and polishing were carried out using a Tegramin (Struers, Netherlands) polishing apparatus using silicon carbide papers for the grinding and diamond pastes for the polishing, with progressive finer steps.

After polishing, the cross-section of each line is inspected by SEM and analyzed. Afterwards, the irradiated samples are immersed in 50% HF for about 2 h to dissolve the amorphous material. After rinsing in demineralized water and drying, SEM inspection of the cross-
sections of formed microchannels (and stacks), at the same locations, is performed.

The lines and stacks of lines are irradiated upon varying the polarization of the incoming laser beam, the repetition rate of the laser with which the pulses are delivered, the energy per pulse and the number of overlapping lines in case of stacks (see Table 1)

4. Results and discussion

Fig. 5 shows the general features of a typical cross-section before (i.e. line) and after (i.e. channel) the wet etching step.

As can be observed from this figure, the shape of the cross-section of the modified line after laser irradiation generally follows the original shape of the laser focal spot. In Fig. 5(a) amorphized sapphire appears darker in the SEM micrographs than crystalline sapphire. As was mentioned in Section 1, the amorphous region is not homogeneous in many cases. That is, the laser-affected volume shows both amorphous regions and crystalline regions. For this reason, the cross-sections of channels obtained after the etching in Fig. 5(b) fail in the model in Fig. 1(c).

The “microexplosions” caused by the absorption of the laser energy \[ E_p \] in a very short time are causing cracking of the crystalline sapphire. As will be shown in the next paragraphs, most of the modified lines are surrounded by cracks which may change in length from few hundreds of nanometers up to millimeters, depending mainly on the pulse energy and the repetition rate. These cracks are not modified in size and morphology by the etching process.

Moreover, at high repetition rates (typically 15 MHz) and high pulse energies (typically 91.4 nJ), the presence of small voids may be observed in the cross-sections of the lines (i.e. prior to etching) as well, see Fig. 6.

Fig. 6. SEM micrograph of a cross-section of a single modified line obtained using a pulse energy \( E_p = 91.4 \text{ nJ} \) and a pulse repetition rate of \( f = 15 \text{ MHz} \). The irradiation was performed along the direction perpendicular to the polarization of the laser beam. At a high energy per pulse and high repetition rates a void can appear near the top of the laser-affected volume. The laser radiated from top to bottom of the picture. The velocity of the stage was \( v = 1 \text{ mm/s} \), the geometrical pulse to pulse overlap (calculated) \( \text{OL} = 99.999\% \).
As mentioned, the linear velocity of the XY stages moving the sample was 1 mm/s, whereas the pulse repetition rate was varied from 0.001 MHz to 15 MHz, corresponding respectively to geometrical pulse to pulse overlaps ranging from 0% (completely separated single modifications) to 99.999%.

4.1. Structures parallel or perpendicular to the polarization of the laser light

The cross-sectional shape of the irradiated and etched structures is affected by the angle between the (linear) polarization direction of the laser radiation [24,28,36–38] and the direction of irradiation (direction along which the stage moves).

Hnatovsky et al. [38] demonstrated that modifying the bulk of fused silica using linearly polarized laser pulses results in periodic structures oriented in a direction perpendicular to the irradiation direction.

Taylor et al. [39] explained the formation, during processing, of periodic structures perpendicular to the polarization of the laser radiation, using the transient nanoplasmonics model. This model theorizes the formation of ionization hotspots during the irradiation by ultrashort laser pulses, which eventually induces plasma. These hotspots may lead to a preferential local ionization of the material. In particular, field enhancement on the boundary of the generated ionized spots facilitates the generation of plasma in the direction perpendicular to the polarization of the laser light.

To study the effect of the orientation of the polarization on the irradiated lines and etched channels, structures were produced both parallel and perpendicular to the polarization of the laser radiation.

Fig. 7. SEM micrographs (top row) and zoomed SEM micrographs (bottom row) of cross-sections of modified lines (non-etched) after irradiation obtained using: (a) $f = 0.1$ MHz and $E_p = 94.5$ nJ, (b) 1 MHz and $E_p = 94.5$ nJ, (c) 5 MHz and $E_p = 91.4$ nJ, (d) 10 MHz and $E_p = 91.4$ nJ. The amorphized parallel nanochannels increase in size and number with increasing pulse repetition rate. In all the pictures the sample is irradiated along the direction perpendicular to the polarization of the laser. The laser radiated from top to bottom of the picture. The velocity of the stage was $v = 1$ mm/s, the geometrical pulse to pulse overlap (calculated) varied from OL = 99.000% for $f = 0.100$ MHz to OL = 99.990% for 10 MHz.

Fig. 8. SEM micrograph of a cross-section of a single irradiated line (non-etched) obtained using $E_p = 91.4$ nJ and $f = 15$ MHz along the direction perpendicular to the polarization of the laser beam before etching. The laser radiates from top to bottom of the picture. The velocity of the stage was $v = 1$ mm/s, the geometrical pulse to pulse overlap (calculated) OL = 99.999%.

If the laser polarization is perpendicular to the scan-direction of the stage, the presence of parallel nanochannels is observed in the focal region (Fig. 1(b)), which are not observed if the irradiation direction is
Fig. 9. SEM micrographs of cross-sections after irradiation of lines and after etching of channels obtained at different repetition rates: (a) $f = 0.001$ MHz, (b) 0.010 MHz, (c) 0.050 MHz, (d) 0.100 MHz, (e) 0.200 MHz, (f) 0.500 MHz, (g) 1 MHz, (h) 5 MHz, (i) 10 MHz, (l) 15 MHz. The laser radiated the samples from top to bottom of the pictures. The velocity of the stage was $v = 1$ mm/s, the geometrical pulse to pulse overlap (calculated) varied from completely detached pulses at $f = 0.001$ MHz to an overlap OL = 99.999% at 15 MHz.
parallel to the polarization direction. These regular amorphized parallel nanolines are observed for pulse repetition rates over 0.1 MHz and are more pronounced when a repetition rate of at least 1 MHz is applied, see Fig. 7.

Upon increasing the repetition rate, an increase in both horizontal and vertical dimension of the cross sections and in the number of nanolines per area is observed. However, at a pulse repetition rate of 15 MHz, a circular shape is observed frequently, see Fig. 8. This spherical shape impedes the propagation of the focused laser beam deeper into the sample. This phenomenon will be explained in detail in Section 4.2.

Objective of this paper is to form hollow/open microchannels and stacks of hollow/open microchannels. Hence, the origin and growth of parallel nanolines/nanochannels are out of the scope of this study. For this reason, in the next sections, only results obtained with the irradiation direction parallel to the polarization of the laser are discussed.

Fig. 10. SEM micrographs of cross-sections after irradiation of lines and after etching of channels obtained using a pulse repetition rate of $f = 1$ MHz at different energies per pulse: (a) $E_p = 457$ nJ, (b) 234 nJ, (c) 94.5 nJ. The laser radiated from top to bottom of the picture. The velocity of the stage was $v = 1$ mm/s, the geometrical pulse to pulse overlap (calculated) $OL = 99.900\%$. 
4.2. Effect of pulse repetition rate on lines and stacks of lines

Fig. 9 shows the effects of pulse repetition rate on the cross-sections of single lines (only irradiation) and channels (after HF-etching of lines). Since the velocity of the stages is constant at 1 mm/s, the geometrical pitch between the laser pulses changes from single isolated pulses (at \( f = 0.001 \text{ MHz} \)) to a geometrical overlap of 99.999% between laser pulses (at \( f = 15 \text{ MHz} \)).

At \( f = 0.001 \text{ MHz} \), the laser pulses are not geometrically overlapping. This is also reflected in Fig. 9(a), where a clear cross-section of the line in the focal region cannot be identified. At 0.010 MHz the geometrical overlap between the laser pulses is over 80% (overlap calculated when
an energy per pulse of 94.5 nJ was used with an effective minimal cross-sectional dimension of 0.6 μm before and after etching). However, when a repetition rate below \( f = 0.100 \) MHz is used (Fig. 9(b) and (c)), the obtained irradiated lines show irregular cross-sections, i.e., amorphous sapphire alternates with crystalline to form an irregular pattern such as the one seen in Fig. 1(c).

This phenomenon may be explained by recrystallization of previously amorphized material. That is, even after the first laser pulse, each subsequent, overlapping laser pulse is irradiating a volume of both crystalline and amorphized material. As was reported by Juodkazis and Misawa [32], this may recrystallize parts of the amorphized material, leaving it non-soluble by the etchant.

Another hypothesis is that the overlap between pulses is not sufficient to cause hollow channels with a constant width, because the material amorphized by previous laser pulses is optically distorting the propagation and profile of the laser beam. This effect will be discussed more in detail in Section 4.4.

For pulse repetition rates ranging from \( f = 0.100 \) MHz to 1 MHz the cross-sections in Fig. 9(d)–(g) show open channels with a constant cross-section along their length. These are the targeted hollow microchannels shown in Fig. 1(a). In these figures, the cross-sectional width increases from 0.6 μm at \( f = 0.100 \) MHz to 1 μm at 1 MHz.

Fig. 9(h), (i), (l) shows that, for pulse repetition rates of 5 MHz and higher, the cross-sections of lines/channels reveal a disrupted and fragmented morphology. At these repetition rates large cracks have a strong effect, detrimental on the propagation and fluence profile of the incident laser beam, and, in turn, on the amorphization of the material.

Moreover, at \( f = 15 \) MHz, the laser-affected regions show a (nearly) circular shape near the top of the laser affected volume and smaller features below it, see Fig. 9(l), as well as Fig. 8. This phenomenon can possibly be associated with the findings of Gamaly et al. [40]. These authors reported the formation of plasma at the apex of the focal spot during the ionization of the material both at high repetition rate and at high pulse energies. This plasma is, most likely, preventing exposure of material below it to the laser radiation.

4.3. Effect of energy per pulse

For pulse repetition rates up to 1 MHz, the sample was irradiated using three pulse energies: 94.5 nJ, 234 nJ, 457 nJ. Below the smallest pulse energy, the material is not affected by the laser (at the mentioned repetition rates), whereas at the highest pulse energy it is not possible to irradiate sapphire without the formation of cracks so large that cause the breaking of the specimen. For pulse repetition rates of \( f = 5 \) MHz, 10 MHz and 15 MHz lower pulse energies were applied because of the power limitations of the laser source. At these pulse repetition rates experiments were performed at \( E_p = 18.9 \) nJ, 46.8 nJ and 91.4 nJ.

Fig. 10 shows the effect of the pulse energy on the cross-sections of irradiated lines and etched channels. The shape of the amorphized region deviates from the shape of the focal spot and the cross-sections show multiple foci, with increasing pulse energy. The latter can be associated with Kerr-induced self-focusing. Self-focusing induced by the electro-optic Kerr effect is a change of the refractive index caused by an applied strong electrical field, in this case the laser radiation. This change in the refractive index causes the focal spot to elongate. If the focusing due to the Kerr effect is counterbalanced by a defocusing effect due to the presence of plasma in the focal spot [40], which lowers the refractive index, this may result in spatial focusing/defocusing (multi-foci) along the propagation axis of the laser beam. The power threshold \( P_{\text{crit}} \) above which Kerr effect is triggered can be expressed as [35]:

\[
P_{\text{crit}} = \frac{\lambda_0^2}{2\varepsilon_0 n_2}
\]

where \( \lambda_0 \) denotes the laser wavelength, \( n_0 \) the linear refractive index (\( n_0 = 1.755 \) at 1030 nm [41]) of the material and \( n_2 \) the nonlinear refractive index (\( n_2 = 3 \times 10^{-16} \) cm\(^2\)W\(^{-1}\)) [42]. Hence, \( P_{\text{crit}} \approx 3.02 \times 10^6 \) W, which has the same order of magnitude of the applied peak powers in this work, especially at \( E_p = 234 \) nJ and 457 nJ. This confirms the possible occurrence of self-focusing/multi-foci at these levels of pulse energy.

4.4. Overlapping of the modified lines

For each repetition rate and pulse energy, single lines were overlapped laterally for producing stacks. The number of stacked lines per structure produced is \( 2^n \), with \( n = 1 \) to 8 (see Table 1). However, at high pulse energies, it is often not possible to go above 8–16 lines because of severe cracking that hampers proper irradiation.

A pitch of 50 nm (corresponding to an overlap of 90%) as the lateral shift between the adjacent lines is used. At low pulse energies, it is possible to create stacks of up to 256 lines, although other factors are influencing the formation of fully empty (with no residual crystalline material left) stacks of microchannels.

Accumulated stress inside transparent materials is known to cause birefringence [43,44]. Stress induced by laser processing can facilitate the etching of the material itself [45]. In this case, though, the difference in refractive indices between unprocessed crystalline, stress-affected and amorphous material will most likely cause a distortion of the intensity profile of the incident laser beam. Fig. 11 shows the etched channels surrounded by stress in the crystalline sapphire (and consequent birefringence) made visible by polarized light microscopy. Although this image does not provide quantitative results, it can be concluded that the surrounding stress increases significantly with increasing number of channels.

Cracks additionally interfere with the formation of amorphous material. Cracked material, in fact, is composed of normal crystalline sapphire, voids and stressed crystalline sapphire. It also has, therefore, a mix of refractive indices, which are affecting the focusing of the laser beam. This is, in most cases, having a shielding effect on the crystalline material, which is directly below the crack, thus preventing its amorphization.

Fig. 12 shows an example of this: a crack occurring at about the center of the stacks of microlines was “shielding” the material under-
Fig. 14. SEM micrographs of cross-sections after irradiation of stacks of lines and the structures left after etching obtained at a repetition rate of 0.500 MHz and a pulse energy of 94.5 nJ. The number of single structures overlapped is in order: (a) 1, (b) 2, (c) 4, (d) 8, (e) 16, (f) 32, (g) 64, (h) 128. The laser radiated from top to bottom of the pictures. The velocity of the stage was \( v = 1 \) mm/s, the geometrical pulse to pulse overlap (calculated) \( \text{OL} = 99.800\% \).

Given the step resolution of 50 nm of the setup and because the minimal cross-sectional dimension of single lines composing the stack is in the order of few hundred nanometers, it should be possible to obtain stacks of microchannels with a single and uninterrupted cross-section. However, amorphized sapphire of a previously irradiated line has a lower refractive index compared to the crystalline sapphire \([46]\). This index difference has a deflecting effect on the incoming beam that irradiates the following line. The result is that subsequent lines are often neath: the lines irradiated after/next to the line during which the crack was generated exhibit a smaller cross-section which, in turn, causes an “interruption” in the structure itself.

Fig. 12(c) shows an optical microscopy picture of the same channel (top view, after etching) showing that the crack, which propagated longitudinally, maintained the shielding effect for almost the entire remainder of the irradiated stack.
irradiated at a slightly tilted angle, which yields a separation between adjacent lines (Fig. 13).

Finally, in Fig. 14 an overview is presented of a series of stacks of microchannels produced with a repetition rate of 0.500 MHz and a pulse energy \( E_p = 94.5 \text{ nJ} \), starting from a single channel (Fig. 14(a)) up to 256 overlapping channels (Fig. 14(h)). The shielding effect caused by the cracks is prominent in Fig. 14(g) and (h), while the separation of the channels caused by mismatch of the refractive indices is visible starting from (d) and in varying degrees for every other case. Overall, the results for experiments with more than 64 lines show the presence of large cracks.

5. Conclusions

A study has been performed on the fabrication of microchannels and stacks of microchannels inside the bulk of sapphire. A detailed step-by-step approach regarding the formation of such channels with an analysis of the main factors playing a role on the final morphology was, to the best of our knowledge, missing. The study included the effects of polarization of the light, repetition rate, pulse energy and number of stacked lines on the morphology and appearance of the structures after irradiation and wet chemical etching. The main results found are the following:

- The first part of the investigation regarded the direction of the irradiation: parallel or perpendicular to the polarization of the light. It was found that, if the sample is irradiated along the direction perpendicular to the polarization of the light, the irradiated lines do not show a smooth single amorphized cross-section, but rather a series of vertical amorphized parallel nanolines propagating along the whole length of the channel. Such structures were not in line with the objective of the study; therefore, next experiments were restricted to irradiation parallel to the light polarization.

- With this arrangement, the next phase comprised studying the effect of repetition rate on the obtained structures. It was found that the ideal window of laser irradiation is between 0.100 MHz and 1 MHz. Below this range the structures do not show a single and constant cross-section, but fragmented. Upon using a repetition rate higher than this range, the modified lines also show irregular cross-sections. In fact, they are disrupted and often contain a circular shape on top of the focal region which is shielding the lower/deeper located material and preventing it to be modified.

- The effect of pulse energy was investigated, and it was found that, if a pulse energy of more than 234 nJ for pulse repetition rates of \( f = 0.001 \text{ to } 1 \text{ MHz} \) is used, the focus is split in multiple foci. The splitting is most probably caused by the Kerr-effect and, as a consequence, the cross-sections of the channels after etching are not as targeted.

- Finally, stacks of microlines were studied by varying the number of single lines composing them. In general, the presence of cracks prevent the formation of hollow stacks of microchannels and limits its size to 64 laterally overlapping channels. Moreover a possible non-sufficient overlapping (technically limited by the setup) often causes separation of adjacent channels.

This work demonstrates the possibility of controlling the cross-sectional shape of channels obtained in sapphire using a double-step processing technique based on femtosecond pulsed laser irradiation and selective etching in hydrofluoric acid. It is believed that structures with a hollow, continuous and constant (along the length of the structure) cross-section can be used for microfluidic applications.

Declaration of Competing Interest

None.

CRediT authorship contribution statement

L. Capuano: Conceptualization, Methodology, Validation, Investigation, Data curation, Formal analysis, Writing - original draft, Writing - review & editing, Visualization. R.M. Tiggelaar: Conceptualization, Methodology, Validation, Resources, Writing - review & editing, Supervision. J.W. Berenschot: Conceptualization, Methodology, Validation, Resources, Writing - review & editing, Supervision. J.G.E. Gardeiers: Conceptualization, Methodology, Validation, Resources, Writing - review & editing, Supervision. G.R.B.E. Römer: Conceptualization, Methodology, Validation, Resources, Writing - review & editing, Supervision.

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