Fabrication of High Aspect Ratio Channels in Fused Silica Using Femtosecond Pulses and Chemical Etching at Different Conditions

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In this work we present an in-depth investigation of the direct writing and chemical etching method emphasizing the importance of the parameters of the etchant. Different etching scenarios are examined when 3D structures are written with different laser parameters in the bulk of fused silica samples which are later etched in an aqueous KOH solution. The temperature of the etchant as well as the concentration was varied. The etching rate as well as the selectivity was measured for all of the different cases. Our results demonstrate, that changing the temperature of the etchant only by 30 deg. C can increase the etching rate by several times, however, the selectivity of the etching process deteriorates in this case.
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1. Introduction

Femtosecond laser pulses are widely applied in both industrial and scientific fields [1] when micromachining various materials. The unique light-matter interaction mechanisms lead to decreased thermal damage, better end-result repeatability as well as additional options when tailoring materials which are not possible with long-pulse lasers [2]. Typical (< 300 fs) femtosecond pulses are shorter by two orders of magnitude than the electron–phonon relaxation time for transparent materials (for metals, the relaxation time is of the same order as the pulse duration [3]), thus the laser affected zone is vaporized, or modified faster than the energy can be transferred to the surrounding lattice.

Consequently, due to the high intensity of the focused laser pulses, non-linear absorption mechanisms enable absorption in materials which are otherwise transparent. This enables 3D writing in the bulk of various transparent materials. If the processing conditions are right, when exposed to ultra-short pulse radiation, certain materials (SiO_2, GeO_2, TiO_2-SiO_2, BK glasses and others [4-7]) form nanogratings which are susceptible to chemical etching (HF acid, KOH) and dissolve faster by a few orders of magnitude as compared to the unmodified regions [8,9]. This process of laser assisted etching (LAE) has enabled complicated 3D shape fabrication from transparent materials. Glasses are attractive for scientific and industrial fields due to the high resistivity and low electrical losses, low or adjustable dielectric constant, adjustable coefficient of thermal expansion [10-12] chemical inertness, transparency in the optical and UV wavelength region for some glasses, excellent mechanical properties that can be tailored via the glass composition and manufacturing process[13,14]. Therefore various structures produced from these materials are interesting for a broad range of applications: biomedical, pharmaceutical (microfluidics, optofluidics, μTAS), micromechanics (precise sensors, actuators) [13-16], electronics and etc. [17]

The process of laser assisted chemical etching is realized by focusing (typically with 0.2 – 0.6 NA optics) ultrashort pulses in the bulk of the material [13,18]. The pulses are absorbed by the bound electrons due to multiphoton absorption and subsequent avalanche ionization. The energy that is stored in the electrons is then relaxed through different non-radiative channels which can last up to μs [14] and electron-phonon coupling [14] which happens on a timescale which is longer by at least an order of magnitude as compared to the pulse duration, therefore the energy is deposited in confined regions and modifications are initiated without collateral damage to the surrounding material.

Within the exposed regions, bulk modified areas featuring sub-wavelength periodic structures (sometimes referred to as Type II modifications [13,19] can be realized. The periodic structure consists of varying oxygen rich and depleted regions. The oxygen rich regions are infact nanopores filed with molecular oxygen (O_2) which was extracted from the now oxygen depleted regions. The exact mechanisms for the formation of such a peculiar structure is still a matter of debate, however a few mainstream theories would include: interference of the photons with the generated plasma wave [20], nanoplasma formation due to inhomogeneities of the glass on a nm scale [21], interference of exiton-polariton modes [22] as well as plasmon-polaritons excited at the interface of the modified-unmodified regions within the bulk [23]. In all cases, the nanopores result from a dense subwavelength plasma modulation and subsequent relaxation of self-trapped excitons [20] that generate molecular oxygen [22].

For such a complicated mechanism, sensitivity to parameter value fluctuations exists when such modified areas

are being produced for applicatory purposes. If putting pure laser parameters (pulse energy, wavelength, repetition rate and etc.) aside, the writing parameters (focusing conditions, translation speed, longitudinal pitch, transverse pitch) also influence the outcome when producing complicated 3D structures [24]. Ultimately, when 3D structures are produced in the bulk and etched, the etching step also influences the end quality as well as process throughput especially if the parameters of the etchant (concentration, temperature) are being varied. In this article, we aim to investigate the sensitivity of various parameter values for the LAE method (determine robustness), as well as investigate the outcome of the etching process when varying the conditions (concentration and temperature) of the etchant which was KOH in this case. This examination was done when varying the values of the laser, writing and etching parameters, therefore possible parameter interactions [25] were avoided. We demonstrate that just as the laser and writing parameters influence the process in terms of etching throughput and quality (roughness and produce structure deviation from intended form), the etching parameters produce variations of the results on a similar scale. We also find that at certain etchant settings, it is possible to produce etched out structures faster by 2-3 fold as compared to typical settings that are found in literature [26]. The ability to increase the etching throughput is interesting for various fields especially fast prototyping.

2. Experimental setup

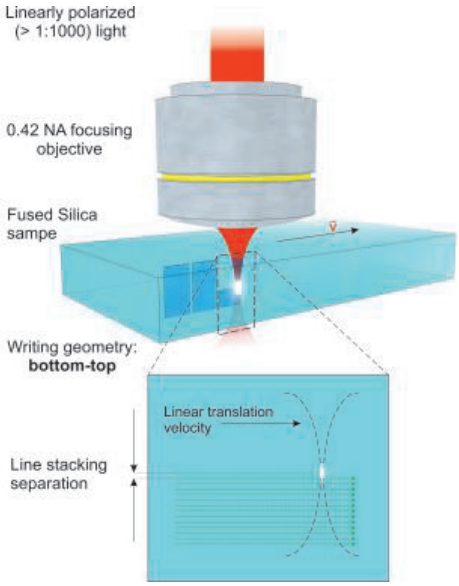

Fig. 1 Writing geometry and components used in the experiments.

In the experiments the influence of laser radiation, writing and etching parameters was investigated for the LAE process. The energy of the pulses (150-750 nJ), longitudinal displacement (1 µm, 5 µm) and translation velocity (0,25-150 mm/s) were varied. For the etching part, a potassium hydroxide solution was used at different concentration settings of 1%, 5% and 35%. The temperature of the solution was also varied and two different settings (80ºC and 110ºC) were used. After the structures are written inside the sample, the samples were etched at the above provided conditions. Moreover, the etching step was divided

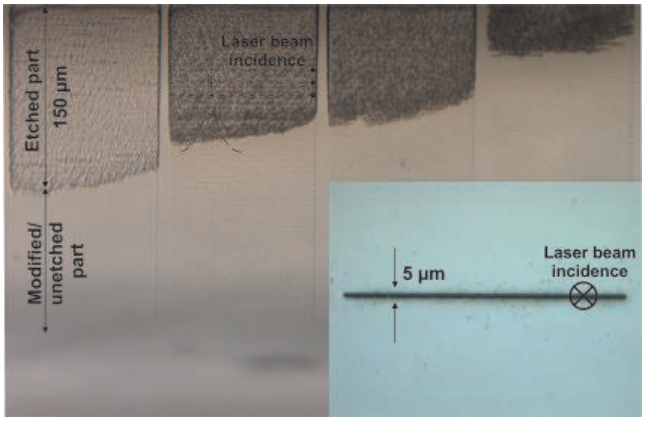

Fig. 2 Images of partially etched out channels after an etching session. A profile view is displayed. Each channel was inscribed with different parameters. The inset shows the entrance of the channel.

into sessions and the total time of etching ranged from 5 minutes to 12 hours, i.e. the etched depth and width was measured after each session, from this the depth to width ratio (D/W) was calculated. The D/W ratio in our case was considered as a measure of etching selectivity.

To fulfill the aim of the experiments, structures of defined geometry (300 µm x 200 µm x 1.5 µm) were inscribed into 1 mm thick fused silica samples and etched. It is worth noting, that in this case 6 parameters are varied and a full factorial [25] approach was used, this equates to approximately 4600 experimental points in total. The cost of doing full factorial experiments is the large amount of time required to perform the experiments, however, in this case, the researcher is sure that the effect of one parameter is not masked due to the setting of the other parameter (parameter interaction are not present). Structures of such dimensions were chosen since these are not overwhelmingly large and it is possible to produce these by the thousands in a reasonable amount of time, moreover, from the etching point of view, these etch at the same rate as larger structures. Therefore these structures can be accounted as a kind of building blocks for larger microfluidic or micromechanical structures.

A femtosecond laser Pharos® (Light Conversion Ltd.) was used operating at 610 kHz (repetition rate), 1026 nm (wavelength) and 260 fs (pulse duration). 610 kHz was the maximum achievable repetition rate of the laser. The higher the repetition rate the higher the overlap of the pulses in space and hence better homogeneity, therefore large repetition rates are preferable for the LAE process. A Glan-Taylor prism was placed before the focusing objective (Mitutoyo NIR 0.42 NA) in order to remove depolarized light that originated due to multiple reflections from HR mirrors. The sample was translated with Aerotech (ABL1500WB) translation stages and controlled with Motion Composer®. The temperature of the etchant was controlled with a custom built KOH reservoir. The reservoir consisted of a stainless steel enclosure filled with KOH which was submerged in a closed container filled with water. The temperature of the water was controlled with a PID controller. Since the water was sealed in a container, it is possible to raise the temperature of the water to above 100 deg. C while the pressure that builds up within the enclosure is less than 4 bars. After each etching step the samples were inspected with an optical microscope (Olympus BX51) and the depth

of the etched channel as well as the width is measured. A typical measurement session is displayed in Fig. 2. When the channels have etched out fully, the sample was separated into two pieces along the writing direction (Fig. 1, along vector **v**). In this case, the channels become fully visible and roughness measurements become possible. The roughness was measured with an optical profilometer (Sensofar PLμ 2300) in topographic mode. The results were analyzed with JMP 14® using parallel coordinates [27] as well as distribution plots.

3. Results and discussion

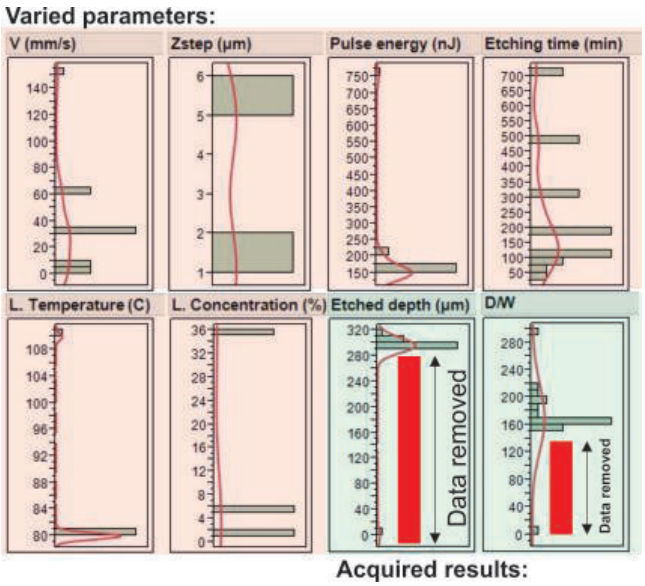

Fig. 3 Density plots of the varied parameter values (top) and acquired results (bottom). Only the channels that have fully etched are displayed and where the D/W ratio is larger than 150 (see text.).

As stated above, multiple experiments were conducted while varying different parameters. Though there have been multiple reports that investigate the influence of individual parameters (pulse energy, translation speed and etc.) [27-29], here we investigate the influence of the parameters for the LAE process while accounting the variability of every other parameter. In this case, a kind of process robustness is estimated, i.e. which parameters influence the process the most and what are the settings of each individual parameter where the process (in terms of etching throughput/selectivity) is least susceptible to change. In addition, it is known, that the etching process is influenced by the temperature of the etchant as well as the concentration of the etchant 30]. When the channels are modified and submerged for etching at different conditions the predictions of the outcomes of etching rate, etched depth and selectivity are not trivial. Therefore we present a deeper look on the influence of the etching parameters. Fig. 3 shows data density plots of the varied parameters and acquired results. In this case, only the successful results are displayed and other experimental points are removed. The unsuccessful results are reproducible in all cases. We attribute the unsuccessful experiments (channels that have not fully etched out even after 12 hours) to disruptive or partial nanograting formations within the modified channel when parameter settings are far from optimal. Since the focused beam is Gaussian, the very top and bottom of the beam produce less absorption in the material as compared to the center. In this case, if the energy of the pulse is near the edge of the working process window, and the translation speed and Zstep are large the areas between consecutive lines may not produce enough exposure to form nanoratings. For these settings, the D/W ratio is always low and etching time may be high. An illustration can be seen in the top right part of Fig. 2 , where an etched line structure appears within the modified channel. Therefore we show only the channels that have etched out fully (the inscribed 300 μm depth channels have etched out fully) and the D/W ratio is greater than 150 which means that the top entrance of the groove is less than 2 μm, which is similar to the dimensions of the focused laser beam. The derivative of each density plot with respect to the value demonstrates sensitivity of each individual parameter given that specific range. From Fig. 3, we see that the majority of experiments with successful results are achievable when the translation speed is set to 30 mm/s. And changing this setting to 120 mm/s will result to a decrease in successful experiments by a factor of 8, i.e. the likelihood of a channel etching out fully and producing a D/W ratio above 150 is reduced by 8 fold. Of course if we were to fix the translation speed 120 mm/s, the density plots would assume a different shape, since parameter interactions terms are present. e.g. if the speed were fixed to 120 mm/s, then perhaps an axial step setting of 1 μm would be required and a successful experiment with 5 μm would not be achievable. However, as a first approximation of l parameter sensitivity, such a representation is adequate. When looking at the axial step density plots, we see that roughly an equal number of counts were registered. Indeed, the axial step (Zstep) seems to not influence the experimental outcome. When working with 0.42 NA focusing conditions and pulse energy values 150-750 nJ, the length (in the Z direction) of the modified area is greater than 5 μm. In this case our results show that as long as the separation (in the Z direction) is not greater than the length of the modified area, there is no significant difference in the results. Such a result is important when talking about fabrication throughput, since structures are produced 5x faster with 5 μm as compared to 1 μm. When talking about pulse energy, the sensitivity to a specific settings is the most important when comparing to the previous two parameters, since the derivative would assume a largest value. This is mainly due to a decrease in etching selectivity when fabricating channels with higher pulse energy settings. This is in good agreement with other parties that have conducted investigations into the effect of pulse energy on the LAE process [26, 28, 29, 31]. We see thatas a parameter, the etching time influences the outcome as well, since the majority of successful experiments were produced after etching for 120-180 minutes. This demonstrates that over-etching results mainly in a decrease of the D/W ratio as well as for a specific parameter combination, a specific amount of etching time will yield the best results. When looking at the etching parameters: temperature and concentration of the liquid etchant, no significant difference in the density plot is visible for concentration (similar results are achievable with all different concentration settings). How-

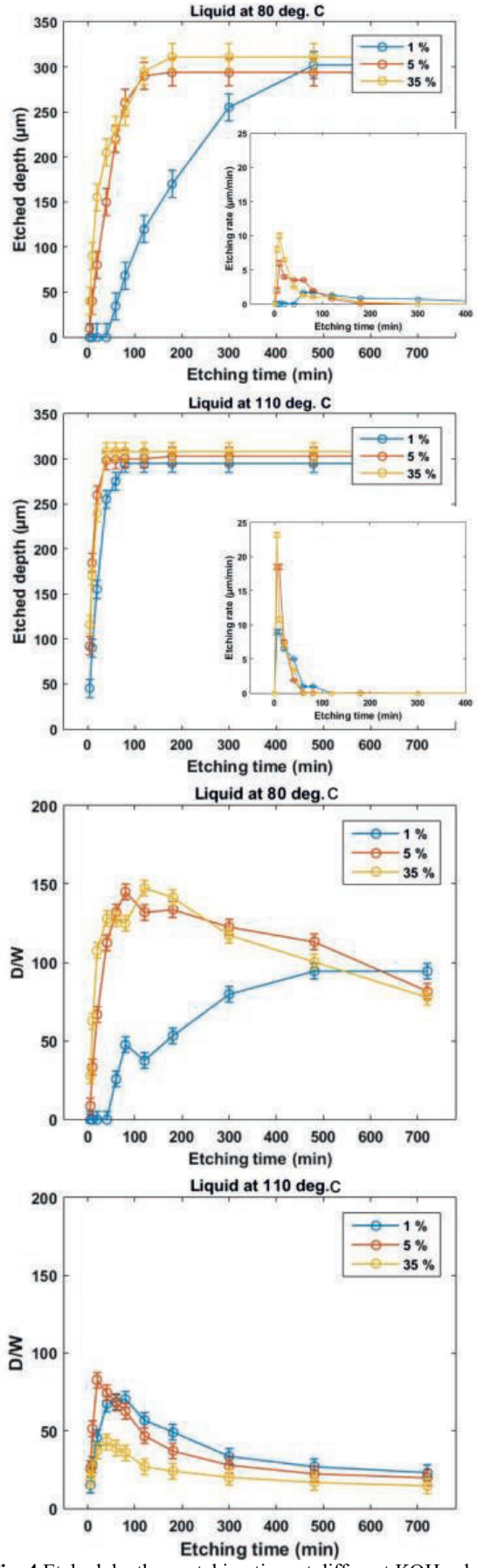

Fig. 4 Etched depth vs. etching time at different KOH solution concentrations and temperature settings and D/W (depth to width) values vs. etching time graphs.

ever the process seems to be sensitive to the temperature of the etchant, in fact, even though the channels etch out relatively fast, it is not possible to achieve D/W values greater than 100 with the 110 deg. C temperature setting at all. In order to demonstrate this better, a deeper investigation into these two parameters was performed.

Fig. 4 shows an etched depth vs. etching time plot for the different concentration settings and the different temperature settings. In this case the parameters: translation speed (v), axial step (Zstep) and pulse energy are fixed at 30 mm/s, 5 µm and 150 nJ respectively. The best results in terms of structure fabrication are achievable with these settings and the variability of the results in this case comes from the etching parameters. At the 80 deg. C setting (top graph) one can notice, that the defined geometry channels (200 µm long and 300 µm deep) etch out fully in the first two hours if the concentration is set to 5 – 35 %, however, it takes nearly 8 hours to fully etch out the structure with the 1 % concentration KOH solution. In this case, the 1 % KOH solution produces an average etching rate of 0.65 µm/min, whereas the 5 % and 35 % solution produce average etching rates equal to 2.42 µm/min and 2.6 µm/min. Clearly, there is a nearly 4 fold difference when etching with 1 % and 5 %. Such a difference is understandable, since the etching rate should scale linearly with concentration, since the amount of ions that react with the SiO_2 sample is greater. However, when moving from 5 % to 35 %, a proportional increase as for the case of 1-5% is not registered, and the average etching rate is only greater by a factor of approx. 1.1. Such a result is attributed to the fact that in all cases, narrow channels are being etched, and the depleted etchant needs to be renewed by diffusion in order to sustain a constant etching rate. Therefore it is believed, that diffusion of the etchant limits the etching rate when the depth of the channels have increased above a certain point. In our case, for the first 50 µm depth (diffusion is fast in this case) the difference in etching rate for the 5 % and 35 % settings is 4 fold.

For the 110 deg. C case, the situation is similar. For the 5 – 35 % concentration settings, the average etching rates are nearly identical at 7.7 µm/min. It is likely, that the difference was masked by the experimental error. For the 1 % concentration setting, the average etching rate was calculated at 3.69 µm/min. When comparing the 80 deg. C case with the 110 deg. C case for all of the different concentration settings, it is worth noting, that moving from 80 deg. C to 110 deg. C produced the largest increase in average etching rate for the 1 % setting. In this case the increase of the average etching rate equals nearly 6, whereas the for the 5-35 % settings, the increase can be evaluated to approximately 3 fold. We attribute this difference again to the insufficient diffusion of the depleted etchant from the channel. Though the etching rate has increased dramatically for the 1 % setting, etching still takes approximately twice as long as compared to the 5-35 % settings. These calculations of relative etching throughputs were calculated from the top two upper graphs of Fig. 4. In the text, average etching rates were addressed, however, the interested reader may find more information regarding the evolution of etching rate as time (and depth) progresses in the insets of the two upper graphs of Fig. 4.

The lower two graphs of Fig. 4 display the evolution of the D/W (depth to width ratio) values over time. In our case, the D/W ratio was regarded as a parameter describing the selectivity as well as quality of the etched channel, i.e. if the D/W ratio is high, then the etched structure will have steep walls and the effect to the unmodified areas is minimal. Moreover, it is worth noting, that the dimensions of the modified area also depend on the writing parameters (energy, translation speed). Two sets of parameters may etch at equal rates, however, if one set of parameters produces wider channels (possibly due to greater exposition or pulse energy) then this set would be regarded as inferior to the one that produces narrower channels given the specific focusing conditions. For the case of the 80 deg. C liquid, as the channel is being etched the D/W values appear similar for the 5 – 35 % concentration settings and peak after roughly 2 hours of etching and start decreasing linearly. This is the time when the structure (300 µm deep and 200 µm long) has etched out fully, when this happens the channel can only get wider as the peripheral areas of the exposed regions are being etched. When looking at the shape of the data points, there is no sign of saturation for the 5-35 % case, therefore it is likely that far better D/W ratio values are achievable given a different geometry (deeper channels) of the structure. More than twice lower D/W values are achievable for the 1 % setting. Such an outcome is a bit surprizing and hints, that possibly the etching rate ratio of the laser modified and unmodified regions is lower in this case as compared to the 5 % or 35 % cases. For the 110 deg. C case the maximum D/W ratio is lower by approx. 1.3, 1.9 and 3 fold for the 1%, 5% and 35 % etchant concentration cases respectively. The lower D/W settings may be explained again by taking into account the diffusion speed of the depleted etchant and related by-products of the reaction. Though at higher temperatures, the diffusion should be faster due to lower viscosity, overall, this is likely to play a minor role since the unmodified regions result in severe exposure as compared to the 80 deg. C case. Theoretical modelling is required to fully explain such behaviour. It is also worth noting, that even though lower D/W ratios are achievable with the 110 deg. C setting, the etching rate is 2-3 fold higher for 110 deg. C liquid setting. And controlling the etching time at different concentration settings it is possible to control the D/W ratio and hence the tapper angle of the produced structures. For some applications, e.g. blind vias fabrication used in the electronics industry [10], a tapper angle might be preferable. Therefore more options for controlling the shape of the inscribed structures are available when simply controlling the etching parameters. Moreover, using higher temperature settings produces larger tapper angles significantly faster. A case might be made that it is also possible to control the tapper angle of the structures by controlling the parameters of writing. Though this is true, e.g. structures written with 750 nJ will have low (< 50) D/W values (large tapper angle) in all cases that are produced due to long etching. The writing throughput will be lower as compared to the set of parameters that produces higher D/W values. This is understandable since the channels etch out the fastest when the modified area has a homogenously modified nanoporous volume with the least amount of discontinuities [14]. Therefore in our case, by controlling the etching throughput and associated diffusivity of the depleted etchant, it is possible to produce various tapper angle shapes while still writing the channels at the optimum throughput. It is worth mentioning, that raising the temperature of the etchant even further should increase the etching rate as well. However, such experiments were not attempted, due to the significantly more complicated etching apparatus. If the water that is kept within the enclosed environment were heated to even larger values (e.g. 150 deg. C), the pressure within the container would reach dangerous levels and special hardware would be required in this case. Another option to increase the temperature even further would be to use a liquid with a high boiling point, Propylene Glycol would be a likely candidate. However a risk of flammability exists. We estimate, that few hundred µm deep structures should etch out in a matter of minutes given even higher KOH solution temperature settings are used, however, pressure build up within the KOH reservoir is also a matter that should be considered and taken with caution.

As stated in previously, the surface roughness of the fully etched channels was measured by breaking the samples along the writing direction (see Fig. 1 along vector v). We observe different roughness values ranging from 50 nm - 1.5 µm RMS. The roughness is dependent on multiple parameters. An in depth investigation will not be presented here, however it is worth noting that the faster translation speed and larger axial overlap generally result in higher roughness of the walls of the channel. In addition, higher pulse energies result to larger roughness values. Such results are expected since it depends on how homogenous was the initial nanoporous modification and the amount of discontinuities, cracks in it. If looking at the influence of the parameters of etching, no considerable differences are visible when changing the temperature of the etchant or concentration. Fig. 5 shows the possible roughness values. Here the RMS roughness of the walls of the channels is plotted against the D/W ratio in order to demonstrate, that different roughness values are achievable with the different concentration settings and the variability is attributed to change in writing parameters. In addition, certain roughness values are interesting for certain applications and highest or lowest is not necessarily the best scenario. What is more, it is possible to achieve the lowest values of roughness (< 100 nm RMS) with the conditions that achieve D/W ratios above 150 as described previously.

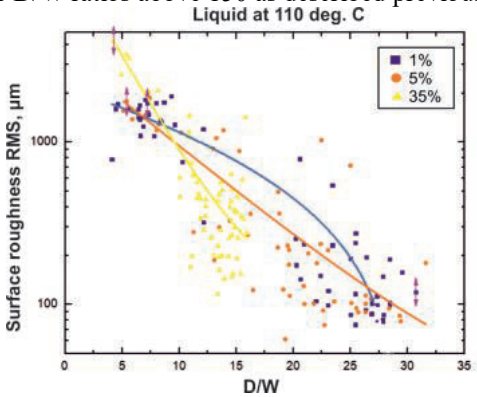

Fig. 5 Measured surface roughness values plotted against the possible D/W ratio. One can notice that different roughness is achievable for different D/W ratio values. The solid lines are eye-guides illustrating that lower roughness typically is achievable with higher D/W ratios.

Conclusion

In this work we have conducted fixed geometry channel fabrication via the laser assisted chemical etching (LAE) method in fused silica samples. The process was investigated when changing the translation velocity, energy of the individual pulses, axial overlap, etchant (KOH) temperature as well as the concentration of the etchant while doing etching sessions of different time. We emphasize the importance of the etching parameters while doing a full factorial study. We demonstrate that the parameters of etching can produce variability in the results (etching throughput, structure quality). With lower (80 deg. C) etchant temperature settings, highest depth to width (D/W) ratio (in our case this was a measure of quality and selectivity) values (up to a factor of 2 as compared to 110 deg. C) are achieved. This is attributed to the insufficient diffusion speed of the depleted etchant from the bottom of the channels since the channels etch out significantly faster (~ 3 fold) with the 110 deg. C setting. Moreover, we demonstrate, that is it possible to control the tapper angle of the fabricated channel simply by changing the etching conditions, which may be interesting for certain applications.

At certain parameter settings: 150 nJ, 5 µm, 30-60 mm/s, 80 deg. C, 5 %, it is possible to achieve fast direct writing (2,25 x 10^{-4} mm^3/s) and etching (5-10 µm/min) conditions, whereas the D/W ratio (selectivity) is high (1:150) and roughness is low (<100 nm). E.g. a 1 mm long and 0.5 mm deep trench is laser fabricated in 3 seconds and etched in ~ 1.5 hours while maintaining the D/W ratio at ~ 1:150. Alternatively a 1 mm long and 0.5 mm deep trench is laser fabricated in 3 seconds and etched in ~ 25 minutes when the D/W ratio of ~ 1:50 is maintained. Fast writing and etching are attributed to non-disruptive (or minimal) nanograting formation at optimal conditions.

Acknowledgements

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